"Effect of catalyst on the synthesis of SiC from rice husk"

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE

REQUIREMENTS FOR THE DEGREE OF

Master of Technology

In

Metallurgical & Materials Engineering

By

Gyanti Prakash Moharana

(209MM1242)

Department of Metallurgical & Materials Engineering

National Institute of Technology

Rourkela

2011

“Effect of catalyst on the synthesis of SiC from rice husk”

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE

REQUIREMENTS FOR THE DEGREE OF

Master of Technology

In

Metallurgical & Materials Engineering

By

GYANTI PRAKASH MOHARANA

(209MM12

Under the Guidance of

Prof.S.C .Mishra

Prof. S. Sarkar

Department of Metallurgical & Materials Engineering

National Institute of Technology

Rourkela

2011

NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA

CERTIFICATE

This is to certify that the thesis entitled, *"Effect of catalyst on the synthesis of SiC from rice husk"* submitted by **Gyanti prakash moharana (209MM1242)** in partial fulfillment of the requirements for the award of **Master of Technology** in **Metallurgical and Materials Engineering** at National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of our knowledge, the matter embodied in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

Prof. S. Sarkar Prof.S.C.Mishra

Dept. of Metallurgical and Materials Engineering

National Institute of Technology, Rourkela-769008

ACKNOWLEDGEMENT

I wish to express my deep sense of gratitude to **Prof. S. C .Mishra**, our guide, for his consistent encouragement, guidance and support to carry out and complete this project, and for giving us an opportunity to work on this project.

I would like to extend my sincere thanks to **Prof.S.Sarkar,** for his guidance and help at different stages of the project.

I am sincerely grateful to **Prof. B. B. Verma**, Head of the Department of Metallurgical and Materials Engineering, NIT-Rourkela, for providing all the necessary facilities in the department.

I am also grateful to **Dr. B. Mallick**, Institute of Physics, Bhubaneswar, for his visionary guidance and analysis techniques highly essential for our project.

I would be highly obliged to extend our thanks to **Mr. Uday Kumar Sahu** and **Mr. Rajesh Patnaik**, for their immense support and help rendered while carrying out my experiments, without which the completion of this project would have been at stake.

I would also like to thank all the staff members of MME Dept., NITR and everyone who in some way or the other has provided us valuable guidance, suggestion and help for this project.

Date:

Gyanti Prakash Moharana(209MM1242)

M. Tech (Metallurgical and Materials Engineering)

National Institute of Technology, Rourkela-769008

“Effect of catalyst on the synthesis of SiC from rice husk”

ABSTRACT

Rice husk is one of the huge amounts of material among them those having no such great use rather creating lots of problem, and its global production is approximately 140 million tons, annually. In the majority of cases much of the husk produced from the processing of rice is either burnt or dumped as a waste. Now days, agricultural waste material are getting a wide exposure to the future generation of material science world. The present study establishes that silicon-based ceramic materials have been prepared from the pyrolysis and heat treatments of rice husks in a N_2 atmosphere. Different Si/C/ N/O based ceramics (SiC, Si_3N_4, Si_2 N_2O and SiO_2) have been produced by heat treatments. The chemical compositions as well as the structure and morphology of these materials were found to be dependent on the temperature and concentration of the Fe introduced before the heat treatment. SEM analysis shows how the morphology of the product changes with change in temperature as well as with the concentration of Fe. Maximum whiskers formation can be observed from the sample of various concentration of Fe treated at 1400°C. At 1450°C, the amount of whiskers is gradually changed to globules.

In the present work, an attempt has been made to synthesize the silicon carbide from rice husk and the effect of Fe catalyst have been studied under the inert atmosphere and this treatment of rice husk will act as a ‘resource’ for energy production and simultaneously avoids its disposal problems. Again it has been found that this route is much more beneficial as compared to other conventional methods of preparation. Since SiC is an important ceramic material by the help of which we can do much more application in future. I have given here much more attention to make the best use of waste materials. Not only by doing so it eliminate the environmental pollution problem but also the best use of waste material can happen. So our society can take the real advantage of modern science and technology.

Keywords : Rice Husk, Pyrolysis, SiC, Scanning Electron Micrographs

. Gyanti Prakash Moharana

Roll No-209MM1242

CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1

INTRODUCTION

1. INTRODUCTION

1.1 RICE HUSK

In recent years, ceramic-matrix or metal matrix composites have become of great interest because of their high temperature strength retention as well as thermal stress resistance coupled with their light weight. As a consequence, different types of Si-based ceramic materials such as SiC, Si_3N_4 and Si_2N_2O are available commercially [1, 2]. For the production of SiC, Si_3N_4 and Si_2N_2O, both the important raw materials SiO_2 and carbon are present in rice husk (RH) with intimate contact and they possess a high surface area. Therefore, it is not surprising that, due to the ease of further processing, RH was reported to be used in the production of SiC, Si_3N_4 and Si_2N_2O by several authors [4-5]. SiC whiskers, useful in reinforcing metal matrix and ceramic matrix composites are already reported to be produced using RH [9-11].We are using RH as a potential source for the production of SiC than other sources because of some specific advantage of it as follows. The major constituents of rice husk are cellulose, lignin, silica and some amount of metallic impurities which vary with the variety of rice, climate and geographic location of growth [6].Again Silica and carbon are naturally occurring in rice husks (RH) and these were used for the production of ceramic materials made of Si, C, N and O as the main constituents.

At present the rice husk is considered as an agricultural waste. Burning has been the primary means of disposal. Not only does burning create pollution problems but the extremely fine silica ash is also toxic and thus constitutes a health hazard. Even careful incineration procedures cannot completely eliminate this airborne silica. Thus, burning with its attendant problems of air pollution and ash disposal has proven to be an unsatisfactory solution. Fortunately, rice husk contains the necessary carbon and silica, intimately dispersed, to provide a nearly ideal source

material for production of SiC, an industrially important ceramic material. Rice husk was first used by Cutler (1973) as a starting material for the production of silicon carbide

Globally, approximately 600 million tones of rice paddy is produced each year. On average 20% of the rice paddy is husk, giving an annual total production of 120 million tones. In the majority of rice producing countries much of the husk produced from the processing of rice is either burnt or dumped as a waste. [7]Rice milling generates a by product know as husk. This surrounds the paddy grain. During milling of paddy about 78 % of weight is received as rice, broken rice and bran .Rest 22 % of the weight of paddy is received as husk. This husk is used as fuel in the rice mills to generate steam for the parboiling process. This husk contains about 75 % organic volatile matter and the balance 25 % of the weight of this husk is converted into ash during the firing process, is known as rice husk ash (RHA).

These husks that are removed during the refining of rice have no commercial interest as such. Another relevant factor is its low cost compared to its large applicability, and its growing demand also reduces the disposal and environmental pollution problems. Rice husk contains silica in hydrated amorphous form and cellulose which yields carbon when thermally decomposed. When such a product is further heated at high temperature (> 1400°C) a reaction occurs between silica and carbon resulting in the formation of SiC. The possible reactions of such a process can be written as

$C(s) + SiO_2(s) = SiO(g) + CO(g)$,

$SiO_2(s) + CO(g) = SiO(g) + CO_2(g)$,

$C(s) + CO_2(g) = 2CO(g)$,

$2C(s) + SiO(g) = SiC(s) + C(g)$,

resulting in the overall reaction

SiO_2 (amorphous) + 3C (amorphous) → SiC + 2CO.

In the plasma furnace the temperature increases rapidly. It has been observed that after 2 min the volatiles are removed. The different varieties of activated silica were also obtained after the calcinations of RH at 600°C followed by the chemical treatment under static and flowing atmospheres of air, argon and oxygen as reported by Yalcin and Serving [12]. RH is a potential source of nanostructures silica having an average particle size of 26nm and specific surface area of 250m^2 g^{-1} as reported by Conradt. Has reported the nano-sized silica from RH with an average particle size of 60nm with specific surface area of 235 m^2 g^{-1} by pyrolysis of RH in air. The present article deals with the preparation of nano-crystallined silica prepared from the pyrolysis of RH, study of different phases of silica as formed and quantitative estimation of silica in the pyrolyzed products from an X-ray diffractometer (XRD) analysis. Energy dispersive spectroscopy (EDS) also confirms the presence of silica and other impurities present in the pyrolyzed products.]. Silica is distributed in the backbone cellulose structure of the rice husk, such that its content is more in the exterior compared with the inner walls [29,30]. Heating the husks produces amorphous carbon as the by-product of carbonization of cellulose, while silica remains unchanged. Pyrolysis of the ash at high tem- peratures and under controlled atmosphere promotes carbothermal reduction of silica and formation of Sack Synthesis of SiC from rice husks was initiated by Culter and Lee in 1975 [29]. Several other researchers attempted various alternative routes to characterize the formation of SiC during carbothermal processes [15,20]. Accordingly, three reactions are responsible for the SiC formation at temperatures from 1200 to 2000°C. These are:

$SiO_2 + 3C \rightarrow SiC + 2CO$

$SiO_2 + 2C \rightarrow Si + 2CO$

$SiO_2 + C \rightarrow SiO\ (g) + CO(g)$

These reactions take place as competing processes at different temperatures and pressures. The same reactions occur at higher temperatures between solid quartz and graphite. In the rice husks with the very high surface area and close contact between the amorphous silica and carbon, SiC forms at lower temperatures (1200-1500). Another advantage of the low temperature formation of SiC is the lack of agglomeration and bonding of particles. Present work aimed at the study. The effect of catalyst in the formation of SiC from rice husk. By using high vaccum newly design mini graphite furnance. In the majority of rice producing countries much of the husk produced from the processing of rice is either burnt or dumped as a waste. [76]Rice milling

generates a by product know as husk. This surrounds the paddy grain. During milling of paddy about 78 % of weight is received as rice, broken rice and bran .Rest 22 % of the weight of paddy is received as husk. This husk is used as fuel in the rice mills to generate steam for the parboiling process. This husk contains about 75 % organic volatile matter and the balance 25 % of the weight of this husk is converted into ash during the firing process, is known as rice husk ash (RHA). This RHA in turn contains around 85 % - 90 % amorphous silica. [77] About 220kg (22 %) of rice husk is produced for every 1000 kg of paddy milled, and when this husk is burnt in the boilers, about 55 kg (25 %) of RHA is generated. India is a major rice producing country, and the husk generated during milling is mostly used as a fuel in the boilers for processing paddy, producing energy through direct combustion and / or by gasification. About 20 million tones of RHA is produced annually. This RHA is a great environment threat causing damage to the land and the surrounding area in which it is dumped. Lots of ways are being thought of for disposing

them by making commercial use of this RHA. RHA acts as a very good insulator. RHA is also used for insulation of molten metal in tundish and ladle in slab caster. The temperature of molten metal in the ladle is around 1400oC and above. When this metal flows from ladle to tundish, the temperature drops to around 1250oC. This reduction in temperature leads to choking and causes breakdown in the slab caster. When this RHA is spread as a coating over the molten metal in the tundish and in ladle, it acts as a very good insulator and the temperature is maintained and does not cool down quickly, hence reducing the breakdown time of the casting. Amorphous Silica, Rice Husk is burnt in controlled temperatures which are below 700oC. This ash generated is amorphous in nature. The transformation of this amorphous state to crystalline state takes place if the ash is exposed to high temperatures of above 850oC. [79] The ash produced after the husks have been burned, (abbreviated to RHA), is high in silica. A number of possible uses are being investigated for this. These uses include

• aggregates and fillers for concrete and board production

• economical substitute for micro silica / silica fumes

• absorbents for oils and chemicals

• soil ameliorants

• as a source of silicon

• as insulation powder in steel mills

• as repellants in the form of "vinegar-tar"

1.2 Rice Husk Properties.

The treatment of rice husk as a ‘resource’ for energy production is a departure from the perception that husks present disposal problems. The concept of generating energy from rice husk has great potential, particularly in those countries that are primarily dependant on imported oil for their energy needs. Rice husks are one of the largest readily available but most under-utilized biomass resources, being an ideal fuel for electricity generation. [13] Rice husk is unusually high in ash compared to other biomass fuels – close to 20%. The ash is 92 to 95% silica, highly porous and lightweight, with a very high external surface area. Its absorbent and insulating properties are useful to many industrial applications. Rice husk ash (RHA) is a general term describing all types of ash produced from burning rice husks. In practice, the type of ash varies considerably according to the burning technique. The silica in the ash undergoes structural transformations depending on the conditions (time, temperature etc) of combustion. At 550°C – 800°C amorphous ash is formed and at temperatures greater than this, crystalline ash is formed. These types of silica have different properties and it is important to produce ash of the correct specification for the particular end use. [13] If a long-term sustainable market and price for rice husk ash (RHA) can be established, then the viability of rice husk power or co-generation plants are substantially improved. Many more plants in the 2 - 5 MW range can become commercially viable around the world and this biomass resource can be utilized largely than at present. [6]

Potential and current uses of RHA are:

 Insulator in the steel industry and as a pozzolan in the cement industry.

 RHA can also replace silica fume in high strength concrete. Silica fume or micro silica is the most commonly used mineral admixture in high strength concrete.

 RHA is used by the steel industry in the production of high quality flat steel. Flat steel is a plate product or a hot rolled strip product, typically used for automotive body panels and domestic 'white goods' products.

 RHA is used, in the manufacture of low cost building blocks and in the production of high quality cement.[6]

1.3 Production of Rice Husk.

The different types of combustion have one common characteristic. They all result in the oxidation of most of the “combustible” portion of the husk while leaving the inert portion. The inert portion is generally called ash or, after gasification, char. The distinction is somewhat blurred. Originally the term “char” referred to the uncompensated residue that had not been taken to a sufficiently high enough temperature to change its state, whereas the term “ash” implied that a higher temperature and change of state had occurred. However, when applied to RHA, the term ash appears to be reserved for all processes apart from gasification irrespective of whether a change of state has occurred.[11] In chemical analyses of husks the term “ash” refers to the chemical constituents of the residual from complete combustion without consideration of the

morphology of the components. The term “ash”, in this study refers to the residual of the particular combustion or gasification process which produced the ash. The fine particulate matter which is carried away from the combustion zone by the flue gas produces fly ash. With stoker and suspension fired boilers this ash is close to 100% amorphous since the crystalline portion of the ash does not seem to carry in the flue gas. Bottom ash is denser than fly ash, and for rice husks tends to be more crystalline than the fly ash. [11]

1.4 Methods of Rice Husk Ash Analysis.

Typically, the ash will contain some unborn components as well as inert Typically, the ash will contain some unburnt components as well as inert components of the husks. The unburnt component is predominantly carbon. It is typically measured by reheating a sample of the ash in an oven. The difference in mass of the sample before and after heating is referred to as the ‘Loss on Ignition’ (LOI). The LOI value is normally the same as the carbon content of the ash. The carbon content of RHA varies according to the combustion process. RHA analyses from a literature search and from analyses performed on RHA material for this study indicate carbon (or LOI) values ranging from 1% to 35%. Typically, commercial RHA combustion appears to result in RHA with 5-7% maximum carbon.[12] The high silica content in the husk may be responsible, in part, for the residual carbon in RHA by ‘cocooning’ the carbon such as to prevent air circulating around it or by bonding to the carbon at the molecular level to form silicon carbide. The silica in the rice husks is at the molecular level, and is associated with water. It occurs in several forms (polymorphs) within the husks. In nature, the polymorphs of silica (SiO_2) are: quartz, cristobalite, tridymite, coesite, stishovite, lechatelerite (silica glass), and opal; the latter two being amorphous. For RHA as a potentially marketable product we need only distinguish between amorphous silica and crystalline silica. From the technical literature, two forms appear to predominate in combustion and gasification. These are lechatelerite (silica glass), an amorphous form, and cristobalite, a crystalline form. SiO_2 can also occur in a very fine, submicron form. This form is of the highest commercial value although it is the most difficult to extract.

The major and trace elements are conventionally expressed as their respective percentage oxides and may not actually be present in this oxide form. SiO_2 is generally determined as ‘total’ SiO_2 since the proportion of crystalline to amorphous silica requires further costly analysis, usually by X Ray Diffraction (XRD). The colour of the ash generally reflects the completeness of the combustion process as well as the structural composition of the ash. Generally, darker ashes exhibit higher carbon content (with the exception of those that may be darker due to soil chemistry/region (see below).[16,17,19] Lighter ashes have achieved higher carbon burnout, whilst those showing a pinkish tinge have higher crystalline (tridymite or cristobalite) content.

1.5 Factors Influencing Ash Properties.

1.5.1 Temperature:

XRD patterns of ash combusted at a range of temperatures from 500oC -1000ºC have shown a change from amorphous to crystalline silica at 800ºC, and the peak increased abruptly at 900ºC. The change from amorphous to crystalline silica at 800ºC was also found in other studies. In Vietnam, a series of experiments using a laboratory oven under conditions designed to simulate the conditions of combustion from a rural facility were carried out .SEM analysis of the ash found that the 'globular' amorphous silica increased in size from 5-10μm to 10-50μm with rising combustion temperatures from 500-600ºC. The transition to completely crystalline silica was complete by 900ºC.

This can be described by below:

: Transition from amorphous to crystalline silica

Note: - quartz converts to _quartz at 573ºC

- quartz converts to tridymite at 870ºC

- tridymite converts to crystobalite at 1470ºC These changes affect the structure of the ash. As such, the 'grindability' and therefore reactivity of the ash is affected since, after grinding, a greater surface area is available for chemical reactions if the ash is to be used as a pozzolan. For the steel industry, more crystalline ash is preferred as this increases its refractory properties.

1.5.2 Geographical Region.

It has been reported that chemical variations in husk composition (and consequently ash composition) are influenced by such things as the soil chemistry, paddy variety and climate. However, only one report of a change in the physical and chemical properties of ash influenced by region was found. A variation in color and trace metal was found in ash from husks burnt in different regions, with ash produced from husks from Northern India resulting in a much darker ash than husks from the US [20,24]. The colour variation was not related to differences in the carbon remaining in the ash, although it is not known the precise regional features that affected the ash. It could be due to the agronomy of the paddy as studies have shown that differences in mineral composition of ash can be attributed to fertilizers applied during rice cultivation, with phosphate having a negative affect on the quality of the ash in terms of its ability to act as a pozzolan . It has also been said that the high K2O found in some ashes could be a consequence of K-rich fertilizers used during the paddy cultivation.

1.6 Review of Influence of Combustion Method on Properties of RHA.

The main factors in the various combustion and gasification processes that determine the type of ash produced are time, temperature and turbulence. These effect all chemical changes that occur

in the combustion process including the way the ash morphology is altered. A study in 1972 compared a range of data for ash composition .The wide range of values was as a result of the variation of purity of the samples and the accuracy of the analytical procedures used. However, since there is no information on different combustion techniques employed in the husk combustion, or analytical techniques used, it is difficult to tell whether any of the reported ranges in chemistries seen could be attributed to particular combustion techniques. Commonly, in the production of highly amorphous ash, low temperatures and fairly long "burn times" are used, as for Mehta's patent. Other work in India has also concentrated on this technique, and has shown how a two-stage process of combustion could control the chemical and physical properties of the resultant ash, increasing its pozzolanic activity by taking the husk through a carbonizing process without "flaming" This type of burning was shown to produce a fine white ash which did not 'carbonize' By comparison, a "normal" combustion process (taking the furnace from room temperature up to the fixed burning temperature, where it was held until combustion was completed) produced a black colored ash. This same study compared the RHA in terms of electrical conductivity and compressive strength tests with concrete. The electrical conductivity is an effective measure of the amorphousness of the ash and showed that the "slow-burn" process produced significantly more amorphous ash.

1.7 RHA Disposal – Negative Benefit

The predominant reason why RHA would only be suitable for disposal is when it residual carbon content exceeds 7%. Based on the power generation technologies examined within the scope of this work, those that seem to result most often in a higher residual carbon content are gasifiers.A plant producing 3MWe using gasifier technology will have multiple units because the application of gasification to rice husks has not been proven in units larger than about 750kWe. The rice husk consumption will be approximately 3.75 tonne/hr and the RHA leaving the gasifiers will be 0.675 tonne/hr. Over the course of a year of operation, the total RHA produced will be 5063 tonnes.[1] Typical disposal cost for RHA for transport and disposal at disposal site range from approximately $5/tonne for local disposal in developing countries where land costs are low, to $50/tonne and higher for disposal to engineered landfills in developed countries. When disposal costs are as low as $5/tonne, the negative cost of producing RHA which has no beneficial use would be $25,315/year or less than 2% of the revenue from electricity.(43) However, when they are $50/tone, the negative cost attributable to disposal could be $253,150/year, almost 20% of the revenue from electricity.

1.8 RHA with Significant Quantity of Crystalline Silica.

Combustion of rice husks, typically in stoker fired boilers, where the ash experiences sustained temperature above 750°C leads to a significant quantity of crystalline silica in the resultant ash. The steel market is preferred for this type of ash. There are no significant additional costs needed to achieve RHA with qualities suitable for acceptance by the steel industry.[28,29,] On the

contrary, stoker boiler technology with over grate feeders for the fuel, and no need to pre-grind the husks, is at the low cost end of the range of equipment choices.

People have already been used Fe, Co, Ni as catalyst and observed that more precipitation of SiC, took place and product gradually turned to globular from whiskers with increase in Fe concentration. The effect of cobalt chloride ($CoCl_2$) treatment on the formation of SiC from burnt rice husks has been studied over a temperature range of 1200 to 1600°C. It has been shown that $CoCl_2$ decreases the crystallization of carbon and silica in rice husk and accelerates the formation of SiC. Formation of SiC whiskers from $CoCl_2$ treated burnt rice husks has been found to be negligible. The untreated burnt rice husks have yielded a maximum quantity of SiC whiskers at 1600°C. Slow heating (at 5C/min) to 1600°C has decreased the reactivity of silica and carbon in the rice husk by increasing their degree of crystallization. No α-SiC formed from the $CoCl_2$ treated burnt rice husks. The synthesis of SiC (both powder and whiskers) was carried out from rice husks with and without the use of catalysts (iron, cobalt and nickel). The introduction of the catalyst increased the reaction rate, the yield becoming up to three times that for the uncatalysed reaction, and increased the proportion of β-phase from 95% to 99%. [43,47]The general behaviour of the three catalysts was very similar, although nickel was the most effective from the point of view of reaction rate, and cobalt in producing larger crystal size. Analysis of the evolution of reaction rate, morphology of the whisker formed, evolution of gases during reaction, crystal size and intermetallic phases, led to a reaction mechanism based on the formation of an M-Si-C liquid phase which is essential for the nucleation and growth of the SiC whiskers. Cobalt has been found to act as a strong catalyst in increasing the formation of total SiC by decreasing the crystallization of carbon black. The formation of SiC whiskers in the presence of cobalt catalyst is found to be low. No alpha SiC has formed in the presence of Co catalyst. With Co catalyst needle-like whiskers are formed. Without any catalyst (a) slow heating has decreased the reactivities of carbon black and rice husk silica by increasing their degree of crystallization and (b) rapid heating has increased the formation of SiC particulates. With Co catalyst rapid heating has been found to be beneficial in increasing the whisker content.

: **Table 1.1 Chemical analysis of rice husks**

Element Analysis	Mass Fraction %
Carbon	41.44
Hydrogen	4.94
Oxygen	37.32
Nitrogen	0.57
Silicon	14.66

Potassium	0.59
Sodium	0.035
Sulfur	0.3
Phosphorous	0.07
Calcium	0.06
Iron	0.006
Magnesium	0.003

:

Compositions	**Mass Fraction (%)**
Silica (SiO2)	80 – 90
Alumina (Al2O3)	1 – 2.5
Ferric oxide (Fe2O3)	0.5
Calcium oxide (CaO)	1 – 2
Magnesium oxide (MgO)	0.5 – 2.0
Sodium oxide (Na2O)	0.2 – 0.5
Potash	0.2
Titanium dioxide (TiO2)	Nil
Loss on Ignition	10 – 20

Table 1.2Compositional analysis of rice husks

Chemical Analysis of Rice Husk Ash

Table 3 shows the chemical composition of rice husk ash. The total percentage of composition of iron oxide (Fe2 O3 = 0.95%), Silicon dioxide (SiO2 = 67.30%) and Aluminum Oxide (Al2O3 = 4.90%) was found to be 73.15%.

Table1. 3. Chemical Composition of Rice Husk Ash

Constituents	% Composition
Fe2O3	0.95
SiO2	67.30
MgO	1.81
Al2O3	4.90

This value is within the required value of 70% minimum for pozzolanas [9]. The value is higher than the value obtained in [5] for acha husk ash (48.36%) and as such the rice husk ash is more pozzolanic. Also this value is less than the 87.55% obtained in [8]. The slight difference in percentage composition might have resulted from the method of preparation of the ash and the species of the rice used.

The loss on ignition obtained was 17.78%. The loss on ignition obtained is less than the value obtained in [5] for acha husk ash (43.57%). This indicates that Acha husk produces greater unburnt carbon compound compared to rice husk. Therefore rice husk is a better material for making pozzolana compared to acha husk. (30)The magnesium oxide content was 1.81%. This satisfies the required value of 4 percent maximum.

1.9 SILICON CARBIDE

Silicon carbide (SiC), also known as carborundum, is a compound of silicon and carbon with chemical formula SiC. It occurs in nature as the extremely rare mineral moissanite. Grains of silicon carbide can be bonded together by sintering to form very hard ceramics which are widely used in applications requiring high endurance, such as car brakes, car clutches and ceramic plates in bulletproof vests. Silicon carbide with high surface area can be produced from SiO_2 contained in plant material [12]. Silicon Carbide is potentially useful such as high temperature structure material, because of its high hardness, high oxidation resistance, good thermal shocking resistance etc. However, the production of high density ceramics by solid state sintering is difficult due to the strong covalent nature of Si-C bond. The solid state sintering of SiC can be performed at high temperature up to 2200^0 C.

1.9.1 NATURAL OCCURRENCES

Moissanite is natural form of Silicon carbide. When the first artificial moissanite reached the jewelry market, it has been regarded as an excellent diamond substitute, with optical properties exceeding those of diamond. Because it has its own unique appearance, it cannot be truly called a diamond stimulant [8] [10]. Its ethical production, however, does make it a popular alternative to diamonds.

Because of its hardness, it is useful for high-pressure experiments (e.g., using diamond anvil cell) competing there with diamond. Large diamonds, used for anvils, are prohibitively expensive. Therefore for large-volume experiments, much cheaper synthetic moissanite is a more realistic choice. Synthetic moissanite is also interesting for electronic and thermal applications because its thermal conductivity is similar to that of diamonds. High power SiC electronic devices are expected to play an enabling and vital role in the design of protection circuits used for motors, actuators, and energy storage or pulse power systems [13] [9] [18].

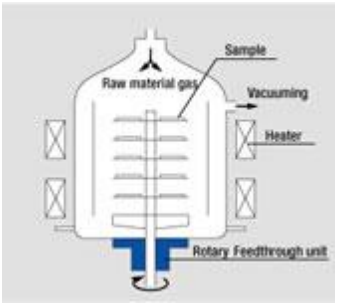
1.9.2 PRODUCTION

Due to the rarity of natural moissanite, silicon carbide is typically man-made. Most often it is used as an abrasive, and more recently as a semiconductor and diamond simulant of gem quality. The simplest manufacturing process is to combine silica sand and carbon in an Acheson graphite

electric resistance furnace at a high temperature, between 1600 and 2500 °C [15]. Fine SiO_2 particles in plant material (e.g. rice husks) can be converted to SiC by heating in the excess carbon from the organic material. he market of ceramics is in constant growth. The employment of this class of materials is strongly encouraged in application involving highly aggressive environments that require resistance to corrosion and to high temperatures (i.e. over 900°C). In addition because of their high hardness and relatively high elastic modulus, they also find uses in high wear applications. Silicon Carbide (SiC) is the leader among various non-oxide ceramics for commercial application: it normally occurs in nature as the mineral moissanite but because of its scarce abundance in nature, SiC is commonly manufactured on a large scale either for use as an abrasive or as a high performance ceramics for semiconductor applications.

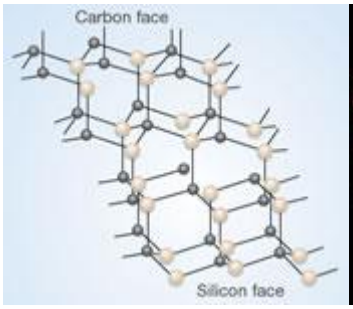
Due to different applications and production methods, the world production of SiC is difficult to evaluate. In 1994 the production amount to 500,000 t/y; today the the market is in constant growth.

Silicon carbide powders can be produced in three principal ways: pyrolysis of silane compounds, direct carbonisation of Si metal, and, of course, carbothermal reduction of SiO_2.



The first method, the so called Chemical Vapour Deposition (CVD) from silane, is expensive and hazardous for the nature of precursor employed, while the carbonization uses a very high cost silicon source. These two methods produce high-purity SiC powders for specific technical applications and they are used for composite materials (i.e. Carbon Fibres) infiltration.

The third method is the cheapest one: it starts from inexpensive silicon dioxide and carbon (or carbon source) that usually react at temperature ranging from 1400-2100 °C to give SiC, which phase depends, as mentioned above, on the synthesis temperature.



Recent developments in technical applications emphasize the role of the starting materials: new applications in the field of ceramic reinforced composites show the improvements of the mechanical properties using nanometer range SiC particles (nanocomposites). Consequently, economical and efficient synthesis routes for nanometer-sized SiC powders are getting more and more important.

Silicon carbide is a high value added material used in a broad range of materials and products: once more cost-effective alternatives to produce it will be available, it could be used even in new markets, while its traditional application will continue to grow in future.

1.9.3 STRUCTURE AND PROPERTIES

Silicon carbide exists in about 250 crystalline forms. The polymorphism of SiC is characterized by a large family of similar crystalline structures called polytypes. They are variations of the same chemical compound that are identical in two dimensions and differ in the third. Thus, they can be viewed as layers stacked in a certain sequence. Silicon carbide exists in about 250 crystalline forms. Alpha silicon carbide (α-SiC) is the most commonly encountered polymorph; it is formed at temperatures greater than 1700 °C and has a hexagonal crystal structure (similar to Wurtzite). The beta modification (β-SiC), with a zinc blende crystal structure (similar to diamond), is formed at temperatures below 1700 °C [8].

Properties	Structure-3C (β)	Structure-4H	Structure-6H (α)
Crystal structure	Zinc blende (cubic)	Hexagonal	Hexagonal
Lattice constants (Å)	4.3596	3.0730; 10.053	3.0730; 15.11
Density (g/cm^3)	3.21	3.21	3.21
Bandgap (eV)	2.36	3.23	3.05
Bulk modulus (GPa)	250	220	220
Thermal conductivity (W/(cm·K))	3.6	3.7	4.9

Table 1.4 Properties of major silicon carbide polytypes

Silicon carbide does not melt at any known pressure [20.. The brown to black color of industrial product results from iron impurities. The high sublimation temperature of SiC (approximately 2700 °C) makes it useful for bearings and furnace parts. Pure SiC is colorless. It is also highly inert chemically. SiC also has a very low coefficient of thermal expansion (4.0×10^{-6}/K) and experiences no phase transitions that would cause discontinuities in thermal expansion There is currently much interest in its use as a semiconductor material in electronics, where its high thermal conductivity, high electric field breakdown strength and high maximum current density make it more promising than silicon for high-powered devices [3] [12].

1.9.4 USES OF SILICON CARBIDE

- Jewelry
- Electric and electronic systems
- Thin film pyrometry and heating elements
- Carborundum printmaking
- Graphene production
- Abrasive and cutting tools
- Structural material
- Automobile parts

CHAPTER 2

LITERATURE SURVEY

2. LITERATURE SURVEY

Fortunately, rice husk contains the necessary carbon and silica, intimately dispersed, to provide a nearly ideal source material for production of SiC, an industrially important ceramic material. Rice husk was first used by Cutler (1973) as a starting material for the production of silicon carbide. Almost all the processes investigated so far involve two process steps i.e. (i) cooking at lower temperature (400–800°C) in a controlled manner to remove volatiles and (ii) reacting the cooked rice husk at high temperature (> 1300°C) to form SiC. Since the rice husk route appears to be promising, much attention has been paid to it (Mansour and Hanna, 1979; Nutt, 1988; Patel, 1991; Ray, 1991; Singh, 1993, 1995; Romera and Reinso, 1996; Moustafa, 1997; Krishnarao, 1998; Padmaja and Mukunda, 1999; Janghorban and Tazesh in 1999; Panigrahi, 2001). Almost all the processes investigated so far involve two process steps i.e. (i) cooking at lower temperature (400–800°C) in a controlled manner to remove volatiles and (ii) reacting the cooked rice husk at high temperature (> 1300°C) to form SiC. These husks that are removed during the refining of rice have no commercial interest as such. Another relevant factor is its low cost compared to its large applicability, and its growing demand also reduces the disposal and environmental pollution problems. since the route of rice husk is more promising much more attention have been paid to it.

U. Kalapathy, A. Proctor, J. Shultz [70] have studied that rice hull ash (RHA), a waste product of the rice industry is its husk. The method based on alkaline extraction followed by acid precipitation was developed to produce pure silica xerogels from RHA, with minimal mineral contaminants. The silica gels produced were heated to 80^{o}C for 12 hr to obtain xerogels. Silica and mineral contents of xerogels were determined by energy dispersive X-ray (EDX) and inductively-coupled plasma (ICP) emission spectrometers, respectively. Xerogels produced from RHA had 93% silica and 2.6% moisture. The major impurities of silica produced from RHA at an extraction yield of 91% were Na, K, and Ca. Acid washing prior to extraction resulted in silica with a lower concentration of Ca (<200 ppm). However, final water washing of the xerogel

was more reactive in producing silica with lower overall mineral content (Na< 200 ppm and K< 400 ppm). X-ray diffraction patterns revealed the amorphous nature of silica xerogel.

T.D. Shen, C.C. Koch, T.L. McCormick, R.J. Nemanich, J.Y. Huang, J.G. Huang, [4] have studied on pressureless sintering of nano-silicon carbide from rice husk. Silicon carbide obtained from direct pryolysis of raw rice husk exhibited very fine microstructure, its sintering behavior may resemble that of nano-silicon carbide. After milling the isostatic compaction done at 250 MPa and then sintered at 1850^{o} C for 6 hours and all analysis have been carried out. It is found that prolonged sintering even at room temperature led at extensive grain growth. On the other hand, SiC from rice husk which although composed of small grain SiC, but those grains are connected together in the form of large agglomerate, therefore, required higher sintering temperature and native porous structure of the rice huskstill remained for some extent after sintering.

C. Srinivasa Rao and G. S. Upadhyaya [83] have studied on the processing and mechanical properties of 2014 and 6061 Al alloy-based power metallurgy composites containing up to 8 ~01% of SIC in either particle or continuous fibre form. For consolidation of the green compacts, liquid phase sintering under vacuum at 635°C was adopted. The addition of reinforcement imparted improved densification such that particulate composites were better densified than the fibrous ones. Relatively higher work hardening rates were observed in fibrous composites than in the particulate ones. The ductility values of obtained porous particulate composites were similar to those of the fully dense once reported in the literature.

Huei-Long Lee, Wun-Hwa Lu and Sammy Lap-Ip Chan [93] have studied on the effects of sintered porosity, volume fraction and particle size of silicon carbide particles (SiC& on the abrasive wear resistance of powder metallurgy (P/M) aluminium alloy 6061 matrix composites. Aluminium alloy 6061 manufactured following the same route was also included for direct comparison of alloy 6061 manufactured following the same route was also included for direct comparison. The results show that the beneficial effect of hard Sic, addition on wear resistance far surpassed that of the sintered porosity in the P/M composites. The wear rates of composites decreased as the amount of Sic, increased. Aluminium alloy composites reinforced with larger SiC, size are more effective against abrasive wear than those reinforced with smaller SiC, size. The wear rates of the P/M aluminium alloy and 10 vol.% SiC, composite were significantly affected by the aging conditions. However, for the 20 vol.% SiC, composite, and for the

composites with large SiC, size, the aging effect on the wear rate of the composites was found to be small.

[**P. K. Mishra, B. C. Mohanty, and B. B. Nayak** [84] have studied that thermal plasma synthesis of silicon carbide from boiler burnt rice husk. Thermal plasma technology has been utilized for synthesis of powder silicon carbide, the burnt rice husk and wood charcoal being processed in a different way. The product has been characterized by chemical analysis, XRD, SEM and also particle size analyser. The yield was 60% which produced both α and β silicon carbide. Based on microstructural study a mechanism has been proposed for formation through liquid phase of SiC.

E. Hellstern, H.J. Fecht, Z. Fu, W.L. Johnson, J. [5] have studied that planetary milling for nano-silicon carbide reinforced aluminium metal matrix composites. High-energy planetary milling was used for mixing aluminium powders with 1 vol.% of silicon carbide (SiC) nano-particles. It was shown that mixing characteristics and reaction kinetics with stearic acid as process control agent can be estimated by normalised input energy from the milling bodies. For this, the additional parameter characterizing the vial filling was determined experimentally. Depending on the ball size, a local minimum in filling parameter was found, laying at 25 or 42% filling of the vial volume for the balls with diameter of 10 and 20mm, respectively. These regions should be avoided to achieve the highest milling efficiency. After a hot compaction, fourfold difference of hardness for different milling conditions was detected. Therewith the hardness of the Al–1 vol.% nano SiC composite could be increased from 47HV0.5 of pure aluminium to 163HV0.5 when milling at the highest input energy levels.

S K Singh, B C Mohanty and S Basu *[31]* The formation of *b*-SiC is observed even in a short time period of 5 min which suggests thermal plasma induced fast reaction due to the high temperature (104 K) associated with it. The reaction time of 20 min appears to be sufficient as there is not much change in the XRD pattern.

Madhumita Sarangi[76] have studied Silica and carbon are naturally occurring in rice husks (RH) and these were used for the production of ceramic materials made of Si, C, N and O as the main constituents. The various "silicon-based" ceramics were produced from the thermal decomposition of rice husks and posterior heat treatment at temperatures varying from 1200–1450°C under a pure nitrogen atmosphere. FeSO4 in various concentrations was introduced to

the decomposed rice husk prior to the heat treatment. The formation of various Si/C/N/O ceramics in general and silicon nitride in particular, were studied with respect to the concentration of FeSO4 (4–10%) as well as temperature (1200–1450°C).The formation of different phases were confirmed by XRD and FT-IR analysis. The present study establishes that silicon-based ceramic materials have been prepared from the pyrolysis and heattreatments of rice husks in a N2 atmosphere. Different Si/C/ N/O based ceramics (SiC, Si3N4, Si2 N2O and SiO2) have been produced by heat treatments. The chemical compositions as well as the structure and morphology of these materials were found to be dependant on the temperature and concentration of the Fe introduced before the heattreatment. SEM analysis shows how the morphology of the product changes with change in temperature as well as with the concentration of Fe. Maximum whiskers formation can be observed from the sample of 6% Fe concentration treated at 1400°C. At 1450°C, the amount of whiskers is gradually changed to globules. The XRD and FT-IR analysis confirms the presence of different phases. Maximum phases were found in the case of 8%Fe treated sample. The possibility of using RH as a precursor for the synthesis of such high temperature structural materials is promising and further work is required for a detailed understanding of their structure and the study of their physical properties for potential applications

Madhumita Sarangi a; S. Bhattacharyya a; R. C. Behera[30] have studied Effect of temperature on morphology and phase transformations of nanocrystalline silica obtained from rice husk. Pyrolysis of rice husk (RH), an agricultural by-product, was studied at different temperatures (700–1100_C) in the presence of air. The changes in morphologies, compositions and phase transformations were studied critically by scanning electron microscopy, energy dispersive spectroscopy and X-ray diffractometry, respectively. Different phases of silica obtained from RH were observed with respect to change in temperatures. Crystallite size and the amount of silica obtained from RH at each temperature were worked out using X-ray diffractometer (XRD) plots. Nano-crystalline silica could be revealed (4.87–18.61 nm) from charred RH at different temperatures (700–1100_C). The amount of silica with respect to other carbonaceous volatile matter was calculated from the XRD plot. Acid treatment (1 : 1 HCl) of RH was also done to remove the metallic impurities. A thermal analysis of raw RH was done to study the decomposition and energy changes in it. From chemical analysis, 18.2% silica was estimated from RRH. The phase transformation takes place in the order: amorphous SiO2 Quartz

!tridymite! cristobalite. Transformation of amorphous silica to crystalline phases occurs gradually at higher calcined temperatures. Crystallite size increases from 4.87 to 18.61nm with increase in temperature from 700 to 1100_C mainly due to agglomeration. Morphology of the RRH, ARH and CRH were studied by SEM, and the presence of silica in the CRH has been confirmed by the elemental analysis carried out during the EDS analysis.

K. Janghorban*, H.R. Tazes[29] have studied Several routes for the production of SiC by pyrolysis of rice hulls have been examined. Under reducing and proper reaction conditions, four distinct processes have been identified; these are, decomposition of silica and cellulose and the formation of SiC particles and whiskers. The efectiveness of sodium silicate as a catalyst on maximization of SiC yield has been discussed. The dependence of SiC yield on the pyrolysis temperature and atmosphere has been described. The reaction products were investigated by XRD, EDS, and SEM. Optimum conditions for the highest SiC yield were determined, and mechanisms for the nucleation andgrowth of SiC whiskers. The rice hulls used in the present work contained 17% silica which theoretically could produce a maximum of 58.5 wt% SiC. Direct pyrolysis at 1400_C and 3 h, under flow of argon, without catalyst (Na2SiO3) and/or at

1400_C and 1 h with catalyst resulted in a near maximum yield of SiC whiskers and particles. Whiskers had several morphologies and were 0.1±1mm thick and 10± 50 mm long. Multistep pyrolysis to 1350_C reduced total SiC, while a slight increase in the SiC whiskers was noticeable. Soaking in dilute solution of sodium silicate enhanced the formation of SiC whiskers and particles; a higher yield was achieved in a shorter time. Concentra- tion of the catalyst and soaking time are crucial para- meters for maximization of the SiC yield.

CHAPTER 3

EFFECT OF CATALYST IN THE SYNTHESIS OF SILICON CARBIDE

A large quantity of the glutinous rice husks is known to have a fibrous material with high silica and carbon contents. Approximately 15-20 wt% of rice husks consists of an ash that is relatively high in silica (> 95% SiO2). This silica has been shown to be a good material for synthesis of pure silicon, silicon carbide, silicon nitride and other silicon compounds. Several investigators have attempted to determine the mechanisms of SiC formation during ablation processes. Three reactions are most likely to occur in the presence of silica and carbon at high temperatures as:

SiO2 (s) + 3 C (s) _ SiC (s) + 2 CO (gas) (1)
SiO2 (s) + 2 C (s) _ Si (s) + 2 CO (gas) (2)
SiO2 (s) + C (s) _ SiO (gas) + CO (gas) (3)

Their thermodynamic calculations indicate that reaction (1) will predominate. The reactions(1), (2) and (3) take place as competing processes at temperature 1300 °C to 1900 °C. Others showed that the effectiveness of Fe, Co and Ni in catalyzing the formation of SiC. Silicon carbide is a commonly used ceramic material with attractive properties such as high strength, stiffness, good wear and corrosion resistance. These properties need for advanced ceramic industries. The aims of the present work were to investigate the optimum conditions of preparing SiC powder and whiskers from the produced silica from acid treated glutinous rice husks; the study includes the analysis of the role of Fe catalysts. People have already been used Fe, Co, Ni as catalyst and observed that more precipitation of SiC, took place and product gradually turned to globular from whiskers with increase in Fe concentration. The effect of cobalt chloride ($CoCl_2$) treatment on the formation of SiC from burnt rice husks has been studied over a temperature range of 1200 to 1600°C. It has been shown that $CoCl_2$ decreases the crystallization of carbon and silica in rice husk and accelerates the formation of SiC. Formation of SiC whiskers from $CoCl_2$ treated burnt rice husks has been found to be negligible. The untreated burnt rice husks have yielded a maximum quantity of SiC whiskers at 1600°C. Slow heating (at 5C/min) to 1600°C has decreased the reactivity of silica and carbon in the rice husk by increasing their degree of crystallization. No α-SiC formed from the $CoCl_2$ treated burnt rice husks. The synthesis of SiC (both powder and whiskers) was carried out from rice husks with and without the use of catalysts

(iron, cobalt and nickel)[.67,80,81] The introduction of the catalyst increased the reaction rate, the yield becoming up to three times that for the uncatalysed reaction, and increased the proportion of β-phase from 95% to 99%. The general behaviour of the three catalysts was very similar, although nickel was the most effective from the point of view of reaction rate, and cobalt in producing larger crystal size. Analysis of the evolution of reaction rate, morphology of the whisker formed, evolution of gases during reaction, crystal size and intermetallic phases, led to a reaction mechanism based on the formation of an M-Si-C liquid phase which is essential for the nucleation and growth of the SiC whiskers. Cobalt has been found to act as a strong catalyst in increasing the formation of total SiC by decreasing the crystallization of carbon black. The formation of SiC whiskers in the presence of cobalt catalyst is found to be low. No alpha SiC has formed in the presence of Co catalyst [88,89] With Co catalyst needle-like whiskers are formed. Without any catalyst (a) slow heating has decreased the reactivities of carbon black and rice husk silica by increasing their degree of crystallization and (b) rapid heating has increased the formation of SiC particulates. With Co catalyst rapid heating has been found to be beneficial in increasing the whisker content. Here we have considered 4% 8%, 10% of Fe as catalyst for the better yield and production of silicon carbide. And we get the better result as compared to other percentage of Fe as catalyst. The present study establishes that silicon-based ceramic materials have been prepared from the pyrolysis and heat treatments of rice husks in a N2 atmosphere. Different Si/C/N/O based ceramics (SiC, Si3N4, Si2 N2O and SiO2) have been produced by heat treatments. The chemical compositions as well as the structure and morphology of these materials were found to be dependent on the temperature and concentration of the Fe introduced before the heat treatment. SEM analysis shows how the morphology of the product changes with change in temperature as well as with the concentration of Fe. Maximum whiskers formation can be observed from the sample of 6% Fe concentration treated at 1400°C. At 1450°C, the amount of whiskers is gradually changed to globules. The XRD and FT-IR analysis confirms the presence of different phases. Maximum phases were found in the case of 8% Fe treated sample. The possibility of using RH as a precursor for the synthesis of such high temperature structural materials is promising and further work is required for a detailed understanding of their structure and the study of their physical properties for potential applications.

CHAPTER 4

EXPERIMENTAL DETAILS

4. EXPERIMENTAL DETAILS

4.1EXPERIMENTAL SETUP REQUIRED

A two step was adopted to prepare SiC directly from rice husk in an indigenously developed pot type extruded high vacuum furnaces using graphite electrode or graphite crucible. A graphite crucible containing the tungsten wire is used to act as bottom of electrode. This small graphite oven consists of small cylindrical graphite oven covered by cap with axial hole through which the out gas will go out. Details of the experimental set up are given below---

a)Graphite crucible with axial hole and cap

A **crucible** is a refractory container used for metal, glass, and pigment production as well as a number of modern laboratory processes, which can withstand temperatures high enough to melt or otherwise alter its contents. Historically, they have usually been made of clay, but they can be made of any material with a higher temperature resistance than the substances they are designed to hold. Ash is the completely unburnable inorganic salts in a sample[46]. A crucible can be similarly used to determine the percentage of ash contained in an otherwise burnable sample of material such as coal, wood, or oil. A crucible and its lid are pre-weighed at constant mass as described above. The sample is added to the completely dry crucible and lid and together they are weighed to determine the mass of the sample by difference.

Figure 4.1—Graphite crucible

Tungsten Wire

Tungsten is the most widely used of the refractory metals. In wire form, it is essential for the production of lighting products such as wire filament, and other goods where its high temperature properties are of use. Among its properties are a melting point of 3410° C, a low coefficient of thermal expansion and low vapor pressure at elevated temperatures, along with good electrical and thermal conductivity.[53]

Important applications of this metal wire are for the production of coiled incandescent lamp filaments, cathode and support structures for power tubes, heating elements for high temperature furnaces and evaporation sources in metalizing processes. Thicker wire sizes, straightened, finish-ground and cut into rod pieces are widely used for glass-to-metal seal lead parts in the lighting and electronic industries.

Tungsten Wire Types

Element produces two types of tungsten wire – Pure and WK **(K-Al-Si doped)** .

Pure tungsten wire is produced in sizes above .020 inches in diameter. Typical usage is for restraightening into rod products and for applications where there is a low alkali content requirement.

WK is wire which has been doped with trace amounts of potassium, causing the wire to exhibit an elongated interlocking grain structure with non-sag properties after recrystallization. WK wire is produced in sizes from less than .001 up to .250 inches in diameter and is used largely for lamp filament and wire filament applications.

Dimensions and Tolerances

Wire diameters are generally expressed in terms of inches or mils (thousandths of an inch). For thin wires from .00 1 inches up to .020 inches in diameter, the diameter of the wire is measured by weight per unit length. That is, the weight expressed in mg of a 200 mm length of wire.

The standard diameter tolerance is ± 3% of the weight measurement. Closer tolerances are available, depending upon the application for the wire product.

To calculate diameter, the equation is:
D (in mils) = .71746 x square root (mg weight/200 mm length)

For thicker wires from .020 inches up to .250 inches in diameter, the inch or mil measurements are used. The tolerances are expressed as a percentage of the diameter. Standard tolerance is ±1.5%.

Other Useful Equations:
meters/kilogram = $102950 / (\text{diameter in mils})^2$
mg/200 mm = $1.9427 \times (\text{dia. in mils})^2$

Tensile Strength

Tensile strength for tungsten wire is usually expressed in grams per milligram weight of a 200

millimeter (or) g/mg/200mm. The normal range of values is from 40 to 110 grams. In general, as tensile strength increases, wire straightness decreases.[34]

Straightness

Wire may be finished in the unstraightened or as drawn condition and is controlled to Elmer's internal standards, In addition, wire can be straightened via mechanical or heat treating processes. Typically, the measure of the straightness of a tungsten wire is obtained by cutting a ten inch long piece of wire and allowing it to lie on a flat clean surface. The maximum deviation from a straight line along its length is the quantitative measure of its straightness. In general, as the degree of straightness increases, tensile strength decreases.

Out of Roundness

Out of Roundness = (Maximum Diameter - Minimum Diameter / Maximum Diameter) x 100

Splits

Longitudinal cracks with a depth of more than 15% of the wire diameter are called splits. The wire is checked for splits with an eddy current split detector. The split level for a given length of wire is typically less than 5%.

QualityandFinish

Wire is free of scratches, kinks and distortions insofar as they are harmful to the intended end use of the product. Surface finish is supplied in the black, as drawn condition, where drawing lubricants and oxides are retained on the surface, or in the cleaned condition by firing in hydrogen or electrochemical etching.

Spooling

Wire is spooled on clean, defect free spools. For very large diameters, tungsten wire is self coiled. Spools are level filled without piling near flanges. The outer end of the wire is properly marked and attached securely to the spool or self coil.

Tungsten Wire Applications and Wire Properties*			
Typical Application	**Description**	**Available**	**Tolerances**

		Diameters		
		From	To	
Halogen Lamp Filaments	Black or Clean	0.001"	0.010"	±1% wt
General Lighting Service (GLS) Incandescent / Fluorescent	Black	0.0008"	0.005"	±1% wt ±3% wt
Electronic Grids and Filaments	Clean	0.005"	0.020"	±1% wt ±3% wt
Heating Elements	Black or Clean	0.005"	0.020"	±3% wt
Redraw Wire	Black or Clean	0.020"	0.100"	±1.5% wt
Electrostatic Precipitators and Medical Devices	Black or Clean	0.008”	0.016”	±1% wt ±3% wt

*** Table 4.1—Tungsten wire and its properties**

c)Black ash prepared from rice husk

The RH was collected from a local rice mill with the husk dimensions of 7–10mm long, 1.5–2.0mm wide and 0.10–0.15mm thick. Dry raw RHs were washed thoroughly with water to remove the adhering soil and other contaminants present in them and then dried in the sunlight for 24 h. These were designated as raw rice husk (RRH). Chemical analysis of these washed RRH was carried out as shown in the flow chart (Figure 1). Acid leaching was performed by treating the RRH with dilute HCl (1 : 1) in distilled water for 1 h. It was then washed thoroughly with distilled water followed by treatment with dilute ammonia solution (10 vol.%) for 1 h to

remove the traces of acid. The treated husk was taken out and washed thoroughly in distilled water followed by drying in air for 24 h.[118]

About 5 g samples of RRH and acid-treated rice husk (ARH) were taken in separate cylindrical alumina crucibles and introduced into a muffle furnace for pyrolysis at different temperatures varying from 700 to 1100_C at an interval of 100_C for 2 h soaking time in static air

d)Vacuum system(10-6 mbar)

A vacuum is a volume of space that is essentially empty of matter, so that gaseous pressure is much less than standard atmospheric pressure. The root of the word vacuum is the Latin adjective vacuous which means "empty," but space can never be perfectly empty. A perfect vacuum with a gaseous pressure of absolute zero is a philosophical concept that is never observed in practice, not least because quantum theory predicts that no volume of space is perfectly empty in this way.[45] Physicists often use the term "vacuum" slightly differently. They discuss ideal test results that would occur in a perfect vacuum, which they simply call "vacuum" or "free space" in this context, and use the term partial vacuum to refer to the imperfect vacuum realized in practice.
The quality of a vacuum is measured by how closely it approaches a perfect vacuum[35-36]. The residual gas pressure is the primary indicator of quality, and it is most commonly measured in units of torr, even in metric contexts. Lower pressures indicate higher quality, although other variables must also be taken into account. Quantum mechanics sets limits on the best possible quality of vacuum. Outer space is a natural high quality vacuum, mostly of much higher quality than what can be created artificially with current technology. Low quality artificial vacuums have been used for suction for millennia.
vaccum has been a common topic of philosophical debate since Ancient Greek times, but it was not studied empirically until the 17th century. Experimental techniques were developed following Evangelista Torricelli's theories of atmospheric pressure. vaccum became a valuable industrial tool in the 20th century with the introduction of the light bulb and vaccum tube, and a wide array of vaccum technology has since become available. The recent

development of human spaceflight has raised interest in the impact of vaccum on human health, and life forms in general.[50]

i)Rotary pump(10-3mbar)

Positive displacement rotary pumps also have their weaknesses. Because of the nature of the pump, the clearance between the rotating pump and the outer edge must be very close, requiring that the pumps rotate at a slow, steady speed. If rotary pumps are operated at high speeds, the fluids will cause erosion, much as ocean waves polish stones or erode rock into sand. Rotary pumps that experience such erosion eventually show signs of enlarged clearances, which allow liquid to slip through and detract from the efficiency of the pump.

Positive displacement rotary pumps can be grouped into three main types. Gear pumps are the simplest type of rotary pumps, consisting of two gears laid out side-by-side with their teeth enmeshed[21-23]. The gears turn away from each other, creating a current that traps fluid between the teeth on the gears and the outer casing, eventually releasing the fluid on the discharge side of the pump as the teeth mesh and go around again. Many small teeth maintain a constant flow of fluid, while fewer, larger teeth create a tendency for the pump to discharge fluids in short, pulsing gushes.

Screw pumps are a more complicated type of rotary pumps, featuring two screws with opposing thread —- that is, one screw turns clockwise, and the other counterclockwise. The screws are each mounted on shafts that run parallel to each other; the shafts also have gears on them that mesh with each other in order to turn the shafts together and keep everything in place.[34-35] The turning of the screws, and consequently the shafts to which they are mounted, draws the fluid through the pump. As with other forms of rotary pumps, the clearance between moving parts and the pump's casing is minimal.

Moving vane pumps are the third type of rotary pumps, consisting of a cylindrical rotor encased in a similarly shaped housing. As the rotor turns, the vanes trap fluid between the rotor and the casing, drawing the fluid through the pump

ii) Diffusin pump(10-2_10-7mbar)

Diffusion pumps use a high speed jet of vapor to direct gas molecules in the pump throat down into the bottom of the pump and out the exhaust. Presented in 1915 by and using mercury vapor, they were the first type of high vacuum pumps operating in the regime of free molecular flow, where the movement of the gas molecules can be better understood as diffusion than by conventional fluid dynamics.(80) Gaede used the name **diffusion pump** since his design was based on the finding that gas cannot diffuse against the vapor stream, but will be carried with it to the exhaust. However, the principle of operation might be more precisely described as **gas-jet pump**, since diffusion plays a role also in other high vacuum pumps. In modern text books, the diffusion pump is categorized as a momentum transfer pump. The diffusion pump is widely used in both industrial and research applications. Most modern diffusion pumps use silicone oil as the working fluid discovered the possibility of using silicone oil in 1928.[[51]]

Figure 4.2 -- Chilled water circulation to keep wall of the furnaces cool.

Advantage

Since the first heating is extremely difficult to achieve in raw vacuum furnances.We have been made a mini graphite furnace in which the required temperature can be achieved in very

small minute. Again the traditional vacuum furnaces those are used can be leaked in high vacuum furnaces and temperature. And cont heating temperature can be maintained by using the mini graphite furnances.To examine the real advantage of this, we just calibrate the pure sample such as Si,Al those having low melting point in vacuum compared to natural melting point. We calibrated here the Si sample.(30) Which is little beat difficult for raw materials calibration by using traditional method of heating.

Calliberation of Si sample

For the standardization value of current and voltage, We kept the Si crystal in a crucible made up of a graphite and introduce it in the heating zone of the furnance.Seal the whole system and vacuum it up to 10-6mbar.Lock the vacuum, so that it can hold for longer period .Start the furnace to heat up to 1414deg centigrade for small time ,5mntas soaking time. Then cool down the system. The filament used here is for heating purpose is Tungsten. No gas flow for the cool down the system. Then release the vacuum and take out the Si crystal. Here We found that standardized value of voltage and current is 20volt and 80amp respectviely.But the time taken to reach the melting point of Si crystal is very small compared to traditional method that is only 5mnt.Finally its come true. Which makes our mini graphite furnaces valuable in the material modification and metallurgical application. When we are preparing or synthesizing some specific type of materials.

Advantage over conventional furnace

We can conclude from the above experimental design of graphite furnances,that this can be well applicable in synthesizing of various type of materials and alternatively the metallurgical modification. As it has lots of advantages over the traditional furnaces those are selling in market, over the plasma heating variable heating value and finally time required is less and less expensive. So this is very much applicable in the development and preparation of various type of materials in the future in material science division specially.

4.2 FORMATION OF SILICON CARBIDE FROM RICE HUSK

1. The RH was collected from a local rice mill with the husk dimensions of 7–10mm long, 1.5–2.0mm wide and 0.10–0.15mm thick. Dry raw RHs were washed thoroughly with

water to remove the adhering soil and other contaminants present in them and then dried in the sunlight for 24 h. These were designated as raw rice husk (RRH).

2. Chemical analysis of these washed RRH was carried out as shown in the flow chart (Figure 1).Acid leaching was performed by treating the RRH with dilute HCl (1:1) in distilled water for 1 h. It was then washed thoroughly with distilled water followed by treatment with dilute ammonia solution (10 vol.%) for 1h to remove the traces of acid. The treated husk was taken out and washed thoroughly in distilled water followed by drying in air for 24 h. About 5 g samples of RRH and acid-treated rice husk (ARH) were taken in separate cylindrical alumina crucibles and introduced into a muffle furnace for pyrolysis at different temperatures varying from 700 to 1100°C at an interval of 100°C for 2 h soaking time in static air, and weight losses were measured after each pyrolysis by an electronic balance.

3. Black ash (BA) and White ash (WA), both the powers were prepared with sufficient quantity (50 g) for further study and synthesis of SiC nanoparticles.

4. A special attachment like graphite furnace with high vacuum attachment was setup. Since the first heating is extremely difficult to achieve in raw vacuum furnances.We have been made a mini graphite furnace in which the required temperature can be achieved in very small minute. Again the traditional vacuum furnaces those are used can be leaked in high vacuum furnaces and temperature. And heating temperature can be maintained by using the mini graphite furnances.To examine the real advantage of this, we just calibrate the pure sample such as Si,Al those having low melting point in vacuum compared to natural melting point. We calibrated here the Si sample. Which is little beat difficult for raw

materials calibration by using traditional method of heating.

Figure 4.3-A graphite crucible with axial hole and cap

5. A special powder container was designed for ball milling the powder in a liquid nitrogen medium.

6. First of the BA prepared from rice husk was taken in the graphite crucible. Then it was placed in high vaccum chamber for four hours(10-^6mbar).The temperature inside the chamber was 800 deg centigrade. for the preparation of sic. For the standardization value of current and voltage, We kept the Si crystal in a crucible made up of a graphite and introduce it in the heating zone of the furnance.Seal the whole system and vacuum it up to 10-6mbar.Lock the vacuum, so that it can hold for longer period .Start the furnace to heat up to 1414deg centigrade for small time ,5mntas soaking time. Then cool down the system. The filament used here is for heating purpose is Tungsten. No gas flow for the cool down the system. Then release the vacuum and take out the Si crystal. Here We found that standardized value of voltage and current is 20volt and 80amp respectviely.But the time taken to reach the melting point of Si crystal is very small compared to traditional method that is only 5mnt.Finally its come true. Which makes our mini graphite furnaces valuable in the material modification and metallurgical application. Heating the husks produces amorphous carbon as the by-product of

carbonization of cellulose, while silica remains unchanged. Pyrolysis of the ash at high tem- peratures and under controlled atmosphere promotes carbothermal reduction of silica and formation of SiC.

Chilled water circulation to keep wall of the furnaces cool.

Figure 4.4-High vacuum furnace at I.O.P Bhubaneswar

4.3 XRD ANALYSIS

The XRD analysis conducted to detect the phases present in the silicon carbide and white ash, which have been made from rice husk were studied by X-ray diffraction using a high resolution PHILIPS X'Pert High Score program system, with Cu - Kα radiation (λ=1.54 A). The phases formed were identified by insidious comparison of the recorded diffraction peaks with the ICDD database. Instrumental broadening was correlated by using FWHM of Silicon standard. X-ray Diffraction scans were conducted for phase present in the produced material.

Parameters	Value
Raw Data Origin	PHILIPS-binary (scan)
Start Position [°2Th.]	10.0100
End Position [°2Th.]	79.9900
Step Size [°2Th.]	0.0200
Scan Step Time [s]	0.6000
Scan Speed	2° (degree) per minute
Scan Type	Continuous
Anode Material	Cu
Voltage applied	30 Kv
Current Flow	20 Ma

Table 4.3 List of parameter used during XRD analysis

4.4 SCANNING ELECTRON MICROSCOPY

The SEM micrographs of composites were obtained using the scanning electron microscope. The images were taken in secondary electron (SE) mode. This analysis was done by a JEOL 6480 LV scanning electron microscope (SEM) equipped with an energy dispersive X-ray (EDX) detector of Oxford data reference system. Microstructural characterization studies were conducted to examine distribution of reinforcement throughout the matrix. This is accomplished by using scanning electron microscope. Silicon carbide produced from rice husk, white ash and sintered composites of Al-1% SiC and pure aluminum samples were metallographically polished prior to examination. Characterization is done in etched conditions. Etching was accomplished using Keller's reagent.

Figure 4.5 JEOL JSM-6480LV scanning electron microscope

Parameters	Value
Detector	Silicon
Accelerating voltage	15.00 (kV)
Process time	5 seconds
Magnification	1000 X, 2500 X, 5000 X, 10000 X, 20000 X

Table 4.4 Parameters used during SEM analysis

CHAPTER 5

RESULT AND DISCUSSION

5. RESULT AND DISCUSSION

5.1 COMPOSITIONAL—ANALYSIS

The compositional analysis of rice husk/sic is done by the weight chemical method, given in the table below.

Element Analysis	Mass Fraction %
Carbon	41.44
Hydrogen	4.94
Oxygen	37.32
Nitrogen	0.57
Silicon	14.66
Potassium	0.59
Sodium	0.035
Sulfur	0.3
Phosphorous	0.07
Calcium	0.06
Iron	0.006
Magnesium	0.003

Compositions	Mass Fraction (%)
Silica (SiO2)	80 – 90
Alumina (Al2O3)	1 – 2.5
Ferric oxide (Fe2O3)	0.5
Calcium oxide (CaO)	1 – 2
Magnesium oxide (MgO)	0.5 – 2.0
Sodium oxide (Na2O)	0.2 – 0.5
Potash	0.2
Titanium dioxide (TiO2)	Nil
Loss on Ignition	10 – 20

5.2 XRD – ANALYSIS

The degree of crystallization of silica increases with increase in Fe concentration, but carbon crystallization decreases. At 4% Fe loading ,the formation of sic peak is more intense than for the 6% and 8% Fe concentration. The oxygen presense in the core of the element is responsible for the intense peak of sic during formation. Again crystallite size increases with increase in temperature mainly due to agglomeration.The phase analysis of silicon carbide (SiC) and white ash, which have been produced from rice husk. The figures shown below are the XRD plots of both SiC and white ash. In the plot SiC all the peaks are sharper, i.e. it represents the silicon carbide is in crystalline form and crystal sizes have been calculated by the help of modified Scherrer formula and crystal size found in the range of 118nm - 50nm. During phase analysis, it is found that all the sharper peaks are of SiC. But But, in the plot of white ash, there are one sharper peak of silicon oxide, i.e. material is almost amorphous. The particle size of white ash is found in the range of 207nm – 35nm.

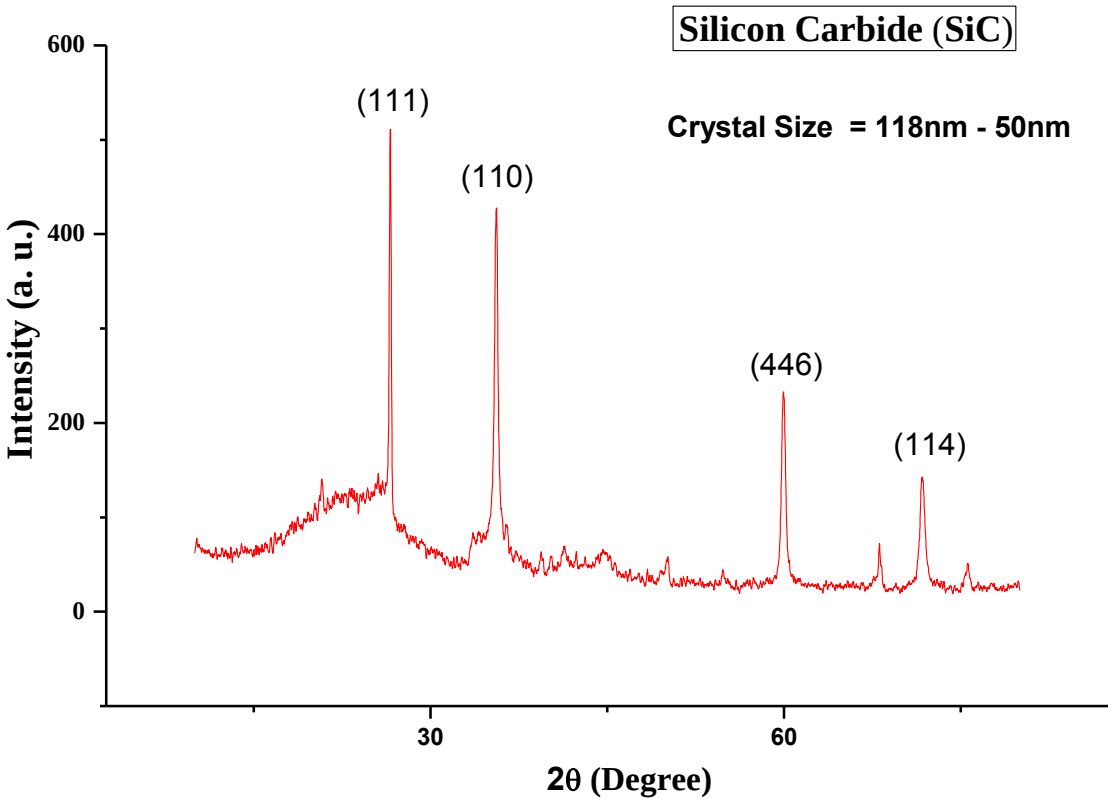


Figure 5.1 XRD plot of SiC produced from rice husk

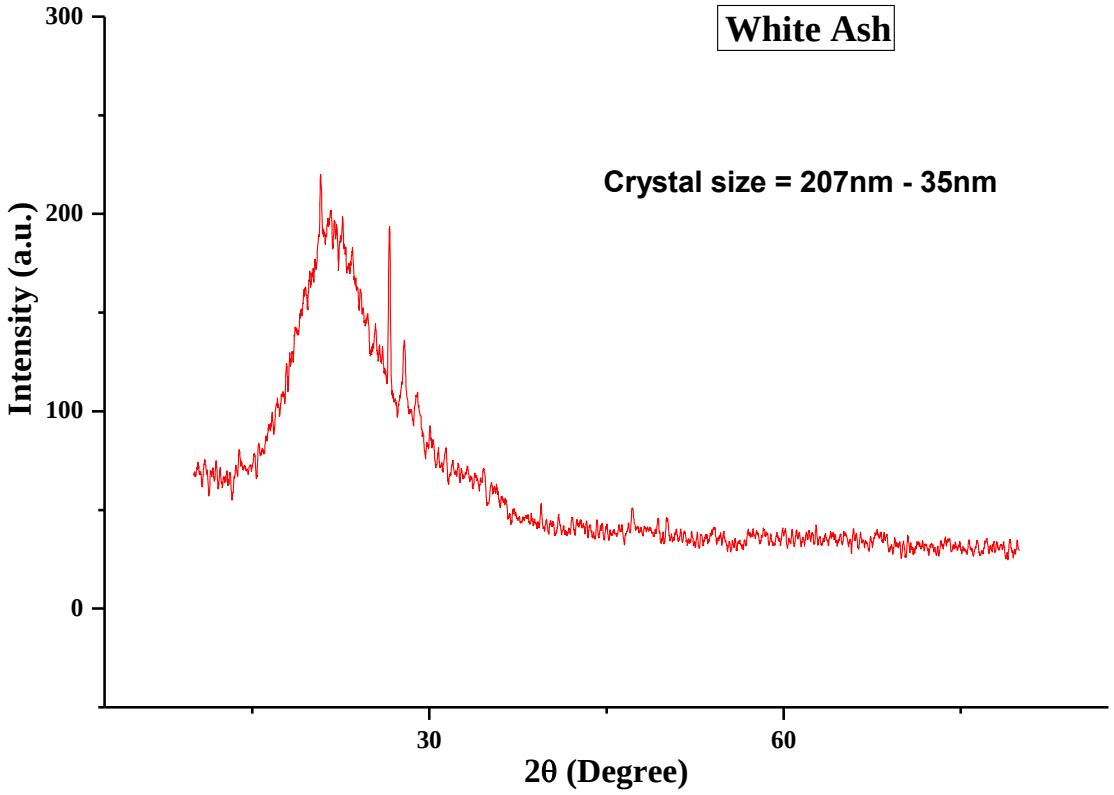


Figure 5.2 XRD plot of white ash produced from rice husk

5.3 SCANNING ELECTRON MICROSCOPY

Micrograph of silicon carbide (SiC) and white ash which has been produced from rice husk is observed under scanning electron microscope, and elemental analysis has been done with EDS.

5.3.1 SILICON CARBIDE FROM BLACK ASH

The formation of SiC crystals are shown in figure 5.3. From the figures it is observed that, the morphology of the product changes with change in temperature as well as Fe concentration, shown in fig. 5.3(b) & fig.5.3(c) . The formation of whiskers of the product started at BA +Fe(4%). As the concentration of Fe increases to 6% the quantity of whisker formation also increases. It was been reported that, the role of iron catalyst in the formation of SiC is significant (76) The liquid phase will act as a sink for silicon, carbon and a source of SiC and Si_3N_4 precipitation. Therefore more precipitation of SiC has taken place with increase in Fe concentration and gradually forms globular shape from whisker type.

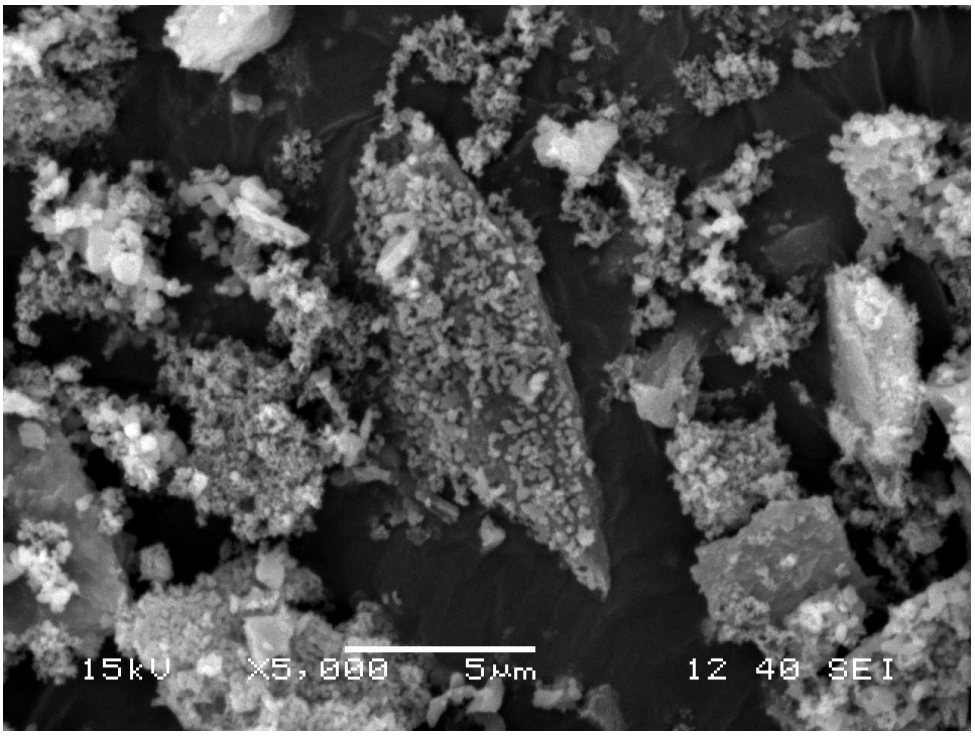


Figure 5.3(a) Microstructure of silicon carbide particles (from black ash).

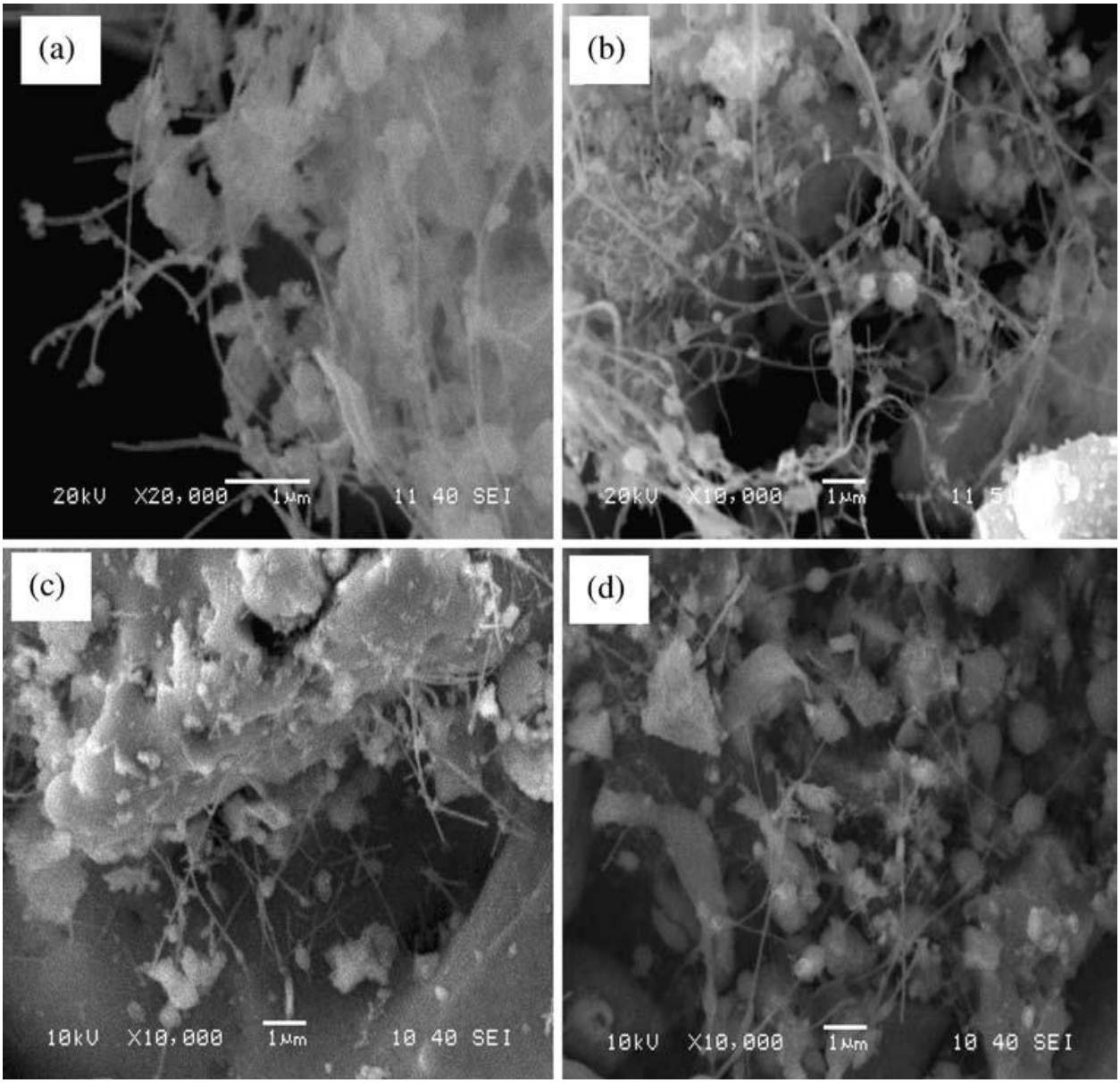


Figure 5.3(b) SEM micrographs of BA+Fe samples a) 4% Fe b) 6% Fe(c) 8% Fe d) 10% Fe; fired at 1400°C/1 h/N_2

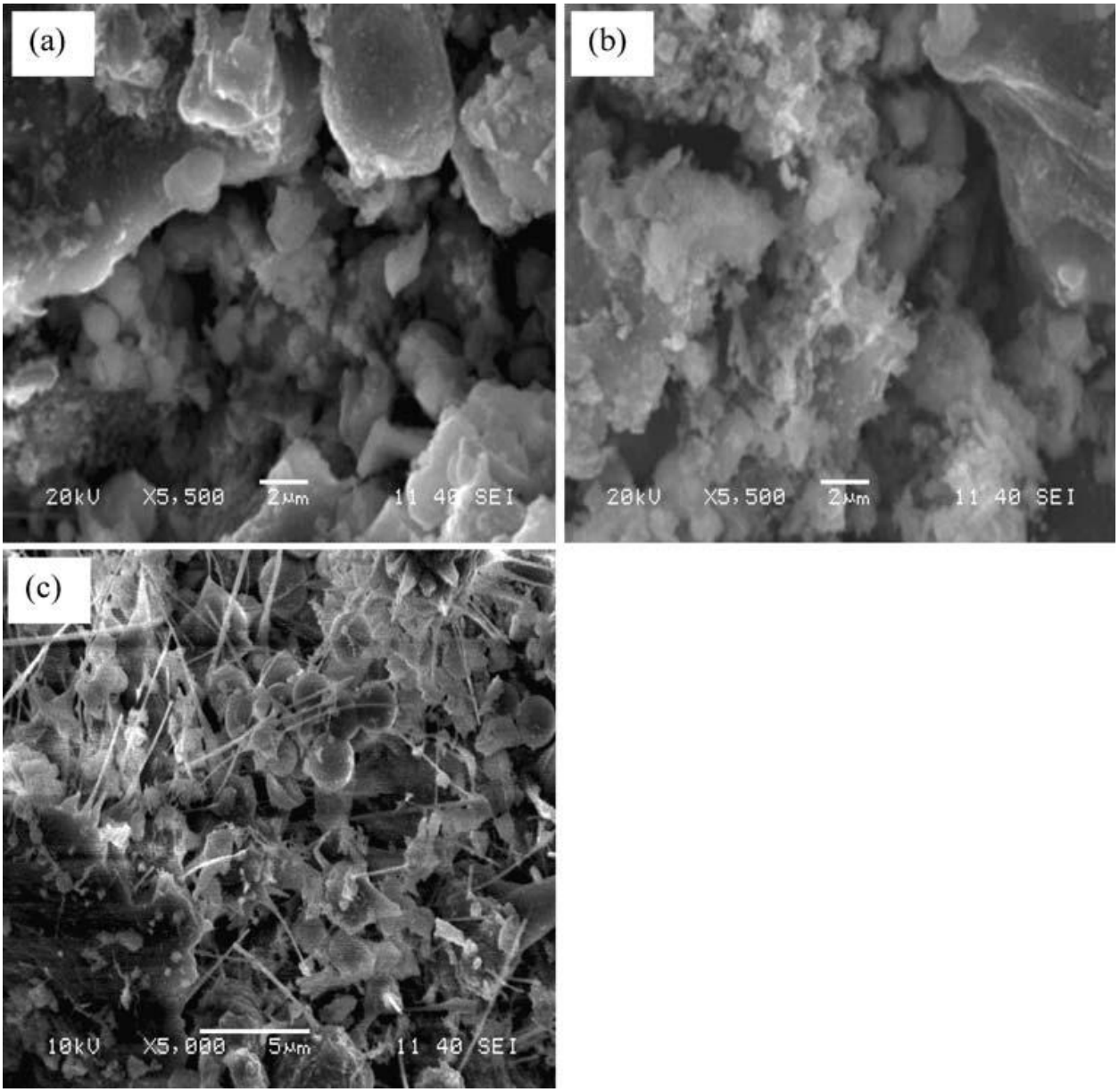


Figure 5.3(c) SEM micrographs of BA+Fe (8%) fired at a) 1200°C, b) 1300°C and c) 1450°C for 1 hr in N_2 atmosphere

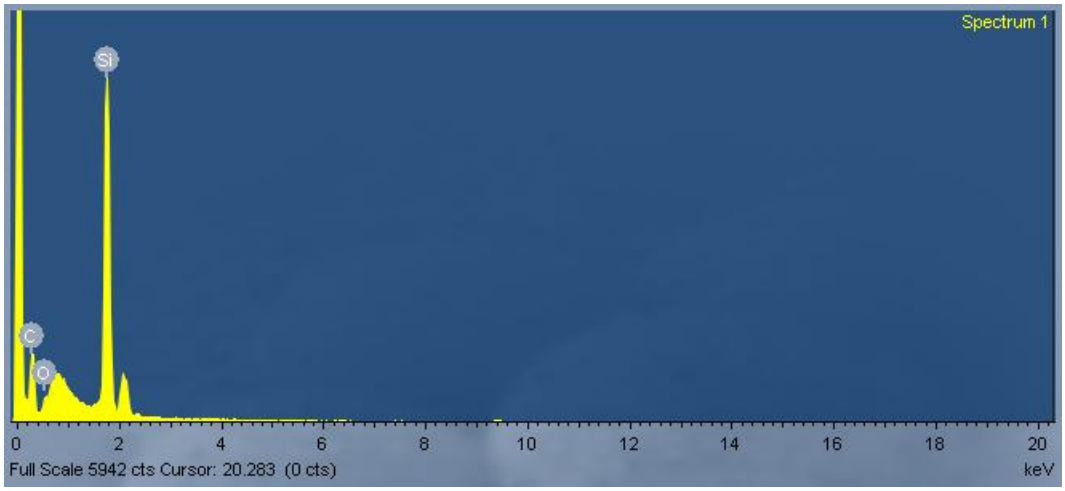


Figure 5.4 EDX pattern of silicon carbide particle

Element	App	Intensity	Weight%	Weight%	Atomic%
	Conc.	Corrn.		Sigma	
C K	11.49	0.3700	57.95	1.19	74.38
O K	1.76	0.5344	6.14	1.09	5.92
Si K	21.02	1.0922	35.90	0.96	19.71
Totals			100.00		

Table 5.1 Compositional analysis of silicon carbide particle

The EDX analysis showing that SiC particle mainly consist of carbon, oxygen and silicon as main constituents.

5.3.2 STRUCTURE OF WHITE ASH

The microstructure of white ash that has been prepared from rice husk by heating it at 800 deg centigrade under the presence of static air is shown in fig.5.5. It can be seen that, with increase in temperature the shape of the product become more globular and increase in particle size also. There is also variation in composition, as evidenced from EDS analysis (table 5.2).

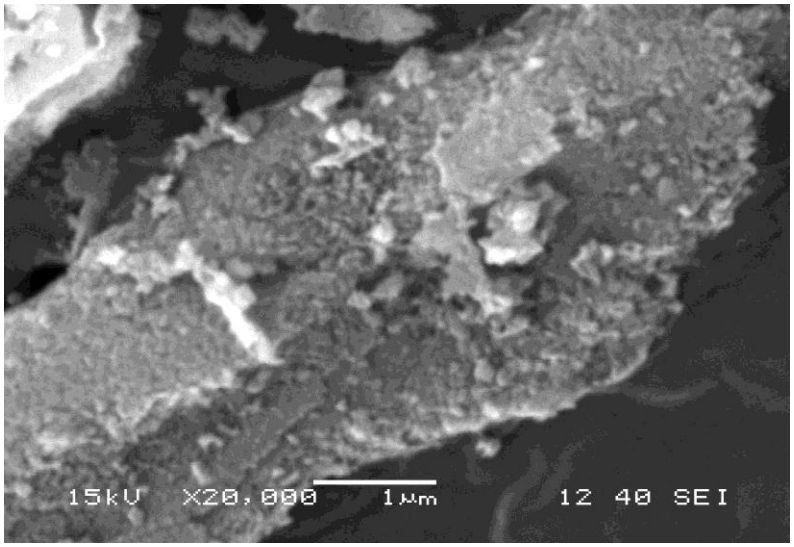


Figure 5.5(a) Microstructure of white ash obtained at 700^0C.

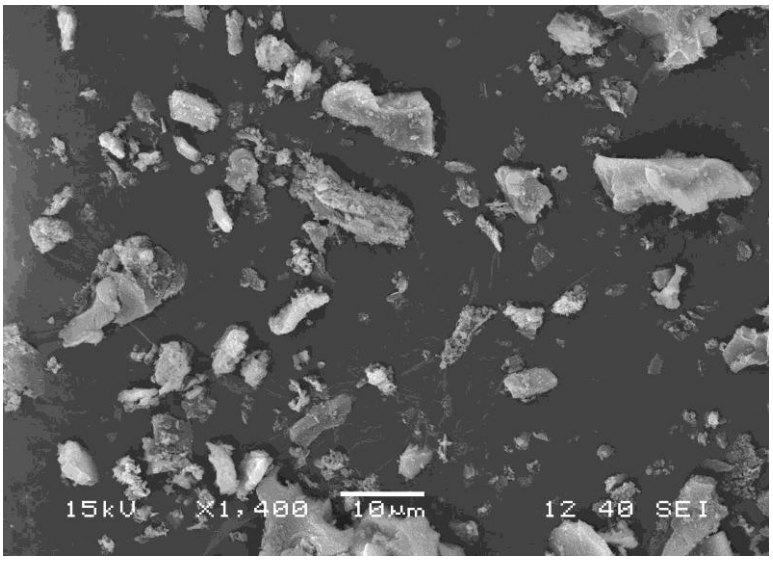


Figure 5.5(b) Microstructure of white ash obtained at 750^0C.

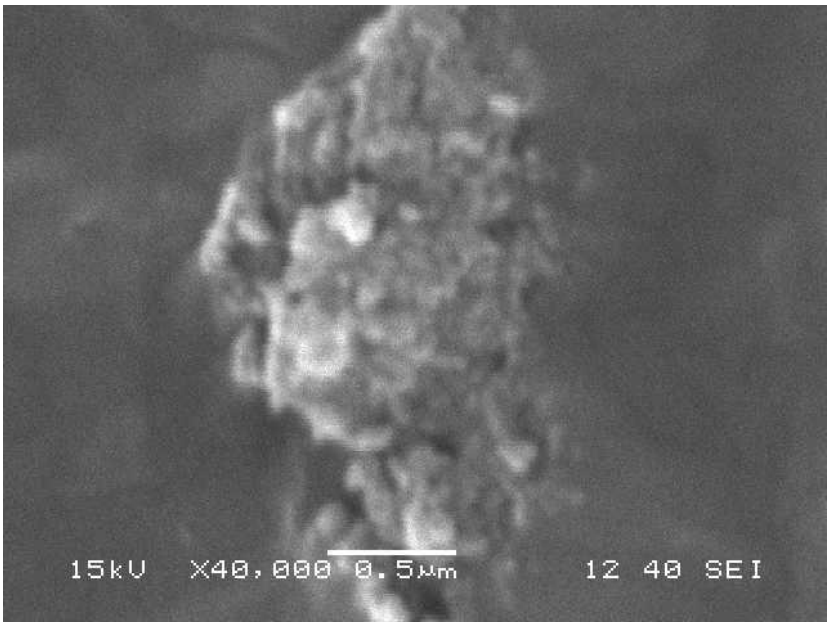


Figure 5.5(c) Microstructure of white ash obtained at 800^0C .

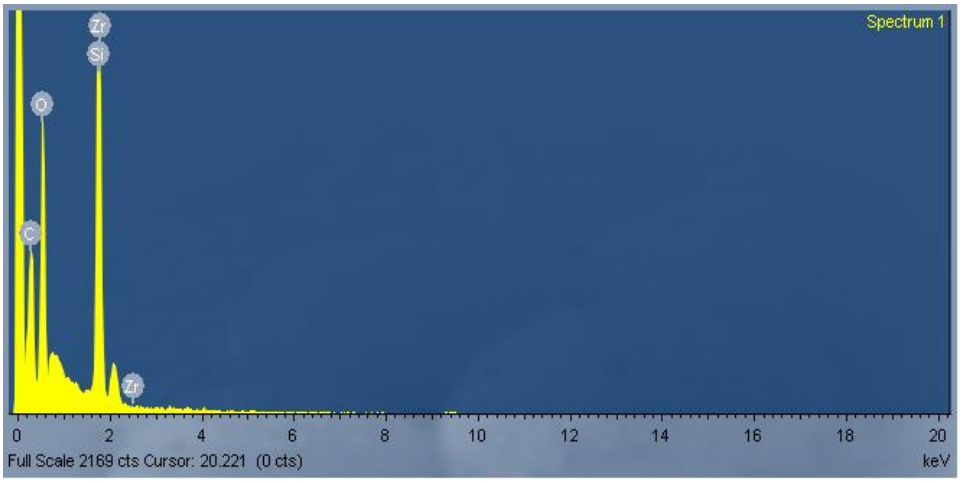


Figure 5.6 EDX pattern of white ash

Table 5.2 Compositional analysis of white ash

Element	App	Intensity	Weight%	Weight%	Atomic%
	Conc.	Corrn.		Sigma	
C K	12.86	0.4956	30.91	1.49	41.94
O K	33.96	0.8764	46.12	1.19	46.98
Si K	14.78	1.0138	17.36	0.50	10.07
Zr L	3.25	0.6905	5.61	0.55	1.00
Totals			100.00		

As the carbon percentage in black ash is more than that of whitw ash, the former is used for processing SiC.

CHAPTER 6

CONCLUSION

6.1 CONCLUSIONS

The following conclusion may be drawn from the present work:

If more be the concentration of Fe(%)more will be the globular of the shape of the product

The silicon carbide (SiC) powder can be synthesized from rice husk.

The prepared SiC powder is almost crystalline particle is appeared.

The produced white ash powder is almost amorphous and some crystalline particle of silicon oxide appeared.

The crystal size of prepared SiC powder is found to be in 118-50 nm range.

6.2 FUTURE WORK

The following points may be concluded for future plan of work:

Silicon carbide produced from rice husk can be scaled up to 100g for the further analysis.

As prepared silicon carbide powder can be kept in liquid nitrogen, alone then it can be mechanically ball milled for different timing and it's TEM, XRD, SEM and other characterizations can be done.

As prepared silicon carbide powder can be mechanically alloyed with aluminum/stainless steel/copper or any other metal/alloys and it's TEM, XRD, SEM and other characterizations can be done to find out the nano-metric dispersion size distribution of SiC in the matrix.

Above mentioned alloyed milled powders can be cold compacted at different pressure ranges, 200-600 MPa.

Sintering can be done under different atmosphere like hydrogen, nitrogen or argon etc. and their impact on sintering density and densification parameter and sintering density can be further improved up 99% by hot compacting/hot isostatic pressure (HIP) method.

Oxidation resistance and corrosion resistance tests under different atmosphere can be carried out.

Above mentioned sintered pellets can be subjected to tensile, compression and impact, hardness at high temperature tests.

White ash having silica, produced from the process can be used for aluminum matrix composite using stir-casting route, the produced MMC can be further characterized and mechanical behavior like tensile strength, compressive strength, impact strength tests can be done.

CHAPTER 7

REFERENCES

REFERENCES

1. B.S. Murty, S. Rang Nathan, *Int. Mater. Rev.* 43 (1998) 101.
2. J. He, J.M. Schoenung, *Mate.Sci.Eng* 336 (2002) 274.
3. R.B. Schwarz, W.L. Johnson, *Phys.Rev.Lett.* 51 (1983) 415.
4. T.D. Shen, C.C. Koch, T.L. McCormick, R.J. Nemanich, J.Y. Huang, J.G. Huang, J. *Mater.Res.* 10 (1995) 139.
5. E. Hellstern, H.J. Fecht, Z. Fu, W.L. Johnson, J. *Appl Phys.* 65 (1989) 305.
6. H.J. Fecht, E. Hellstern, Z. Fu, W.L. Johnson, *Metall Trans.* 21 (1990) 2333.
7. J. Eckert, J.C. Holzer, C.E. Krill III, W.L. Johnson, J. *Mater Res.* 7 (1992) 1751.
8. H.J. Fecht, *Nanostruct Mater.* 6 (1995) 33.
9. D. Oleszak, P.H. Shingu, J. *Appl.Phys.* 79 (1996) 2975.
10. R. Kalyanaraman, S. Yoo, M.S. Krupashankara, T.S. Sundarshan, R.J. Dowding, *Nanostruct.Mater.* 10 (1998) 1379.
11. S. Hwang, C. Nishimura, P.G. Mccormick, *Mater Sci.Eng.* 318 (2001) 22.
12. X. Zhang, H. Wang, M. Kassem, J. Narayan, C.C. Koch, J. *Mater.Res.* 16 (2001) 3485.
13. X. Zhang, H. Wang, M. Kassem, J. Narayan, C.C. Koch, *Scr.Mater.* 46 (2002) 661.
14. X. Zhang, H. Wang, R.O. Scattergood, J. Narayan, C.C. Koch, *Mater.Sci.Eng.* 344 (2003) 175.
15. T.D. Shen, C.C. Koch, *Acta Mater.* 44 (1996) 753.

16. B.L. Huang, R.J. Perez, E.J. Lavernia, M.J. Luton, *Nanostruct.Mater.* 7 (1996) 67.

17. C.C. Koch, *Nanostruct.Mater.* 9 (1997) 13.

18. F.J. Humphreys, M. Hatherly, *Recrystallization and Related Annealing Phenomena,* Pergamon, Oxford, UK, 1996.

19. R V, Krishnarao, R V Mahajan and R V Kumar T J 1998 v *J. Euro. Ceram. Soc.* 18, 1998 **,**147.

20. N A L Manosur and S B Hanna 1979 *Trans. J. Br. Ceram.Soc.* 78 132.

21. S.K Singh,K.M Parida B.C Mohanty and S.B Rao *Reac.Kinet.Catal. Lett.* 54 ,1995, 29.

22. S.F Moustafa,M.B Morsi andA..S El-Din *Can. Metall. Quar.* 36 1997 355

23. S.R Nutt *J. Am. Ceram. Soc.* 71,1998, 149.

24. G. Padmaja and P.G Mukunda *J. Am. Ceram. Soc.* 82, 1999, 1393.

25. M.R Panigrahi and R.P Overand , 1997 in *Proceedings of the third biomass conference of the American* .

26. A.K Ray ,G Mohanty and A. Ghose *J. Mater. Sci. Lett.* 10,1991, 277

27. F.J.N Romera and F.R Reinso *J. Mater. Sci.* 31,1996, 779

29 .K.Janghorban, H.R Tazesh, *Ceramic international* 25 (1999), 7.

30 M .Sarangi, S Bhattacharya, R.C Behera Phase transformation 82 (2009), 377.

31 S.K Singh ,B.C Mohanty, S.Basu *Bull. Mate Sci.* 25, 6,(2002) 561.

32 B.S. Murty, S. Rang Nathan, *Int. Mater. Rev.* 43 (1998) 101.

33 J. He, J.M. Schoenung*, Mate.Sci.Eng* 336 (2002) 274.

34 R.B. Schwarz, W.L. Johnson, *Phys.Rev.Lett.* 51 (1983) 415.

35 T.D. Shen, C.C. Koch, T.L. McCormick, R.J. Nemanich, J.Y. Huang, J.G. Huang, J. *Mater.Res.* 10 (1995) 139.

36 E. Hellstern, H.J. Fecht, Z. Fu, W.L. Johnson, J. *Appl Phys.* 65 (1989) 305.

37 H.J. Fecht, E. Hellstern, Z. Fu, W.L. Johnson, *Metall Trans.* 21 (1990) 2333.

38 J. Eckert, J.C. Holzer, C.E. Krill III, W.L. Johnson, J. *Mater Res.* 7 (1992) 1751.

39 H.J. Fecht, *Nanostruct Mater.* 6 (1995) 33.

40 D. Oleszak, P.H. Shingu, J. *Appl.Phys.* 79 (1996) 2975.

41 R. Kalyanaraman, S. Yoo, M.S. Krupashankara, T.S. Sundarshan, R.J. Dowding, *Nanostruct.Mater.* 10 (1998) 1379.

42 S. Hwang, C. Nishimura, P.G. Mccormick, *Mater Sci.Eng.* 318 (2001) 22.

43 X. Zhang, H. Wang, M. Kassem, J. Narayan, C.C. Koch, J. *Mater.Res.* 16 (2001) 3485.

44 X. Zhang, H. Wang, M. Kassem, J. Narayan, C.C. Koch, *Scr.Mater.* 46 (2002) 661.

45 X. Zhang, H. Wang, R.O. Scattergood, J. Narayan, C.C. Koch*, Mater.Sci.Eng.* 344 (2003) 175.

46 T.D. Shen, C.C. Koch, *Acta Mater.* 44 (1996) 753.

47 B.L. Huang, R.J. Perez, E.J. Lavernia, M.J. Luton, *Nanostruct.Mater.* 7 (1996) 67.

48 C.C. Koch, *Nanostruct.Mater.* 9 (1997) 13.

49 F.J. Humphreys, M. Hatherly, *Recrystallization and Related Annealing Phenomena,* Pergamon, Oxford, UK, 1996.

50 R V, Krishnarao, R V Mahajan and R V Kumar T J 1998 v *J. Euro. Ceram. Soc.* 18, 1998 **,**147.

51 N A L Manosur and S B Hanna 1979 *Trans. J. Br. Ceram.Soc.* 78 132.

52 S.K Singh,K.M Parida B.C Mohanty and S.B Rao *Reac.Kinet.Catal. Lett.* 54 ,1995, 29.

53 S.F Moustafa,M.B Morsi andA..S El-Din *Can. Metall. Quar.* 36 1997 355

54 S.R Nutt *J. Am. Ceram. Soc.* 71,1998, 149.

55 G. Padmaja and P.G Mukunda *J. Am. Ceram. Soc.* 82, 1999, 1393.

56 M.R Panigrahi and R.P Overand , 1997 in *Proceedings of the third biomass conference of the American* .

57 A.K Ray ,G Mohanty and A. Ghose *J. Mater. Sci. Lett.* 10,1991, 277

58 F.J.N Romera and F.R Reinso *J. Mater. Sci.* 31,1996, 779

59 S.K Singh ,L. Stachowicz , S.L Girshick and E.Pfender *J. Mater. Sci. Lett.* **12 1993,**659.

60 S.K Singh , K.M Parida , B.C Mohanty and S.B Rao . *Reac.Kinet.Catal. Lett.* 54, **1995,**29.

61 A. Jain, T.R. Rao, S. Sambi, and P. Grover, *Energy and chemicals from rice husk,*. 7 (1994),. 285..

62 A. Chakravety, P. Mishra, and K. Banerjee, *Investigation of thermal decomposition of rice husk*, 94 (1985),. 267..

63 A. Sharma and T.R. Rao, *Kinetics of pyrolysis of rice husk*,. 67 (1999),. 53..

64 D.M. Ibrahim and M. Helmy, *Crystallite growth of rice husk ash silica*, 45 (1981), 79.

65 M. Andreoli, G.T. Luca, and E.S.M. Seo, *Characteristics of rice husks for chlorination reaction,*. 44 (2000),. 294.

66 R.V. Krishnarao, J. Subrahmanyam, and T.J. Kumar, *Studies on the formation of black particles in rice husk silica ash,* J. Eur. Ceram. Soc. 21 (2001),. 99

67 T.H. Liou, *Preparation and characterisation of nano-structured silica from rice husk, Mater. Sci. Eng* 364 (2004), 313.

68 R.K. Vempati, S.C. Musthyala, M.Y.A. Mollah, and D.L. Cocke, *Surface analyses of pyrolysed rice husk using scanning force microscopy,* 74 (1995)1722.

69 V.P. Della, I. Kuhn, and D. Hotza, *Rice husk ash as an alternate source for active silica production,* 57 (2002), 818..

70 U. Kalapathy, A. Proctor, and J. Shultz, An *improved method for production of silica from rice hull ash,* 85 (2002),. 285.

71 S.R. Kamath and A. Proctor, Silica *gel from rice husk ash: Preparation and characterisation,*

72 www.vacuum.com

73 Wikipedia of diffusion pump

74 B.B Panigrahi , G.G Roy and M.M Godkhindi *Br. Ceram. Trans.* 100 ,2001, 29.

75 R P Overend and E Chornet *New York: Elsevier Science*,. 79.

76. M. Sarangi. Effect of Iron Catalyst in Synthesis of Rice Husk vol-1 2009 pp 103-106

77. S.K Singh ,L. Stachowicz , S.L Girshick and E.Pfender *J. Mater. Sci. Lett.* **12 1993,**659.

78. S.K Singh , K.M Parida , B.C Mohanty and S.B Rao . *Reac.Kinet.Catal. Lett.* 54, **1995,**29.

79. A. Jain, T.R. Rao, S. Sambi, and P. Grover, *Energy and chemicals from rice husk,*. 7 (1994),. 285..

80. A. Chakravety, P. Mishra, and K. Banerjee, *Investigation of thermal decomposition of rice husk*, 94 (1985),. 267..

81. William D. Callister Jr.**,** John Wiley and Sons, (2008), page-162-702.

82. S.L.Kakani, Amit Kakani, New Delhi**,** New age international publishers (2004), page-593-613.

83. C. Srinivasa Rao and G. S. Upadhyaya, PII:SO261-3069(96)0015-5.

84. P. K. Mishra, B. C. Mohanty, and B. B. Nayak**,** , Institute of Minerals and Materials Technology**,** Bhubaneswar.

85. U. Kalapathy, A. Proctor, J. Shultz**,** ***"***Bioresource Technology 73 (2000) 257-262***.***

86. Bharat B. Panigrahi, Materials Science and Engineering A 460–461 (2007) 7–13.

87. S. R. Kamath, A. Proctor, Cereal Chemistry, (1998), 484-487***.***

88. R.V. Krishnaro, Godkhindi, M.M., 1992. Ceramics Intl. 18, 243-249.

89. A. Chakraverty, Kaleemullah, S., 1991. Energy Consers. Mgmt., 32, 565-570.

90. A.K. Jha , PhD thesis, IIT Kanpur, 1988.

91. S. Sarkar,***",*** National Institute of Technology, Rourkela.

92. L. Lu, M.O. Lai and S. Zhang, "Key Engineering Materials vols. 104-107 (1995) pp. 111-124.

93. Huei-Long Lee, Wun-Hwa Lu and Sammy Lap-Ip Chan, **,** Wear, 159 (1992) 223-231***.***

94. E. Mizuki, S. Okumura, H. Saito, S. Muro, " **,** Bioresources Technology, (1993) 44, 47-51***.***

95. G.Arthur, "Journal of The Institute of Metals, 1954, 83, 329-336.

96. Lauri Kollo, Marc Leparoux, Christopher R. Bradbury, Christian Jäggi, Efraín Carre˜no-

97. Morelli, and Mikel Rodríguez-Arbaizar**,** Journal of Alloys and Compounds 489 (2010) 394–400.

98. Y.H. Tang, Y.F. Zhang, N. Wang, C.S. Lee, X.D. Han, I. Bello, S.T. Lee, Appl. Phys. Lett. 85 (1999) 7981.

99. R.S. Wagner, W.C. Ellis, Appl. Phys. Lett. 4 (1964) 89.

100. E.I. Givargizov, J. Cryst. Growth 20 (1973) 217.

101. M. Morales, C.M. Lieber, Science 279 (1998) 208.

102. X. Duan, C.M. Lieber, Adv. Mater. 12 (2000) 298.

103. M.S. Gudiksen, J. Wang, C.M. Lieber, J. Phys. Chem. B 105 (2001) 4062.

104. C.L. Cheung, A. Kurtz, H. Park, C.M. Lieber, J. Phys. Chem. B 106 (2002) 2429.

105. D. Wang, F. Qian, C. Yang, Z. Zhong, C.M. Lieber, Nano Lett. 4 (2004) 871.

106. Y. Wu, P. Yang, J. Am. Chem. Soc. 123 (2001) 3165.

107. X.M. Meng, J. Liu, Y. Jiang, W.W. Chen, C.S. Lee, I. Bello, S.T. Lee, Chem. Phys. Lett. 382 (2003) 434.

108. C.C. Chen, C.C. Yeh, Adv. Mater. 12 (2000) 738.

109. Y.W. Wang, L.D. Zhang, C.H. Liang, G.Z. Wang, X.S. Peng, Chem. Phys. Lett. 357 (2002) 314.

110. M.H. Huang, Y. Wu, H. Feick, E. Weber, P.D. Yang, Adv. Mater. 13 (2000) 113.

111. H.S. Tsai, G.J. Jaw, S.H. Chang, C.C. Cheng, C.T. Lee, H.P. Liu, Surf. Coat. Technol. 132 (2000) 158.

112. P. Pan, W. Berry, J. Electrochem. Soc. 132 (1980) 3001.

113. S. Veprek, Thin Solid Films 130 (1985) 135.

114. P. Heszler, L. Landstrom, M. Lindstam, J.O. Carlsson, J. Appl. Phys. 89 (2001) 3967.

115. E. Borsella, S. Botti, R. Alexandrescu, I. Morjan, T. Dikonimos-Makris, R. Giorgi, S. Martelli, Mater. Sci. Eng. A 168 (1993) 177.

116. Y.L. Li, Y. Liang, K.S. Xiao, R. Zheng, Z.Q. Hu, Mater. Lett. 22 (1995) 87.

117. S.C. Tjong, H. Chen / Materials Science and Engineering R 45 (2004) 1–88 81

118. Z.Y. Ma, S.C. Tjong, Y.L. Li, Compos. Sci. Technol. 59 (1999) 263.

119. G.L. Messing, S.C. Zhang, G.V. Jayanthi, J. Am. Ceram. Soc. 76 (1993) 2707.

120. M.R. Zachariah, M.I. Aquino, R.D. Shull, E.B. Steel, Nanostruct. Mater. 5 (1995) 383.

121. J. Karthikeyan, C.C. Berndt, J. Tikkanen, J.Y. Wang, A.H. King, H. Herman, Nanostruct. Mater. 8 (1997) 61.

122. H. Hahn, Nanostruct. Mater. 9 (1997) 3.

123. A. Singhal, G. Skandan, N. Glumac, B.H. Kear, Scr. Mater. 44 (2001) 2203.

124. J.H. Yu, J.S. Lee, K.H. Ahn, Scr. Mater. 44 (2001) 2213.

125. W. Chang, G. Skandan, H. Hahn, S.C. Danforth, B.H. Kear, Nanostruct. Mater. 4 (1994) 345.

126. W. Chang, G. Skandan, S.C. Danforth, B.H. Kear, Nanostruct. Mater. 4 (1994) 507.

127. M. Ebelman, Ann. Chim. Phys. 16 (1846) 129.

128. A. Cossa, Il Nuevo Cimento 3 (1870) 228.

129. A. Stock, K. Somieski, Ber. Chem. Ges. 54 (1921) 740.

130. N. Tohge, G.S. Moore, J.D. Mackenzie, J. Non-Cryst. Solids 63 (1984) 95.

131. C.J. Brinker, J.W. Scherer, Sol–Gel Science: The Physics and Chemistry of Sol–gel Processing, Academic Press,

132. Boston, 1990.

133. A.C. Pierre, Introduction to Sol–Gel Processing, Kluwer Academic Publishers, Boston, 1998.

134. B.E. Yoldas, J. Mater. Sci. 21 (1986) 1080.

135. C.J. Brinker, K.D. Keefer, D.W. Schaefer, R.A. Assink, B.D. Kay, C.S. Ashley, J. Non-Cryst. Solids 63 (1984) 45.

136. R.K. Iler, The Chemistry of Silica, Wiley, New York, 1979.